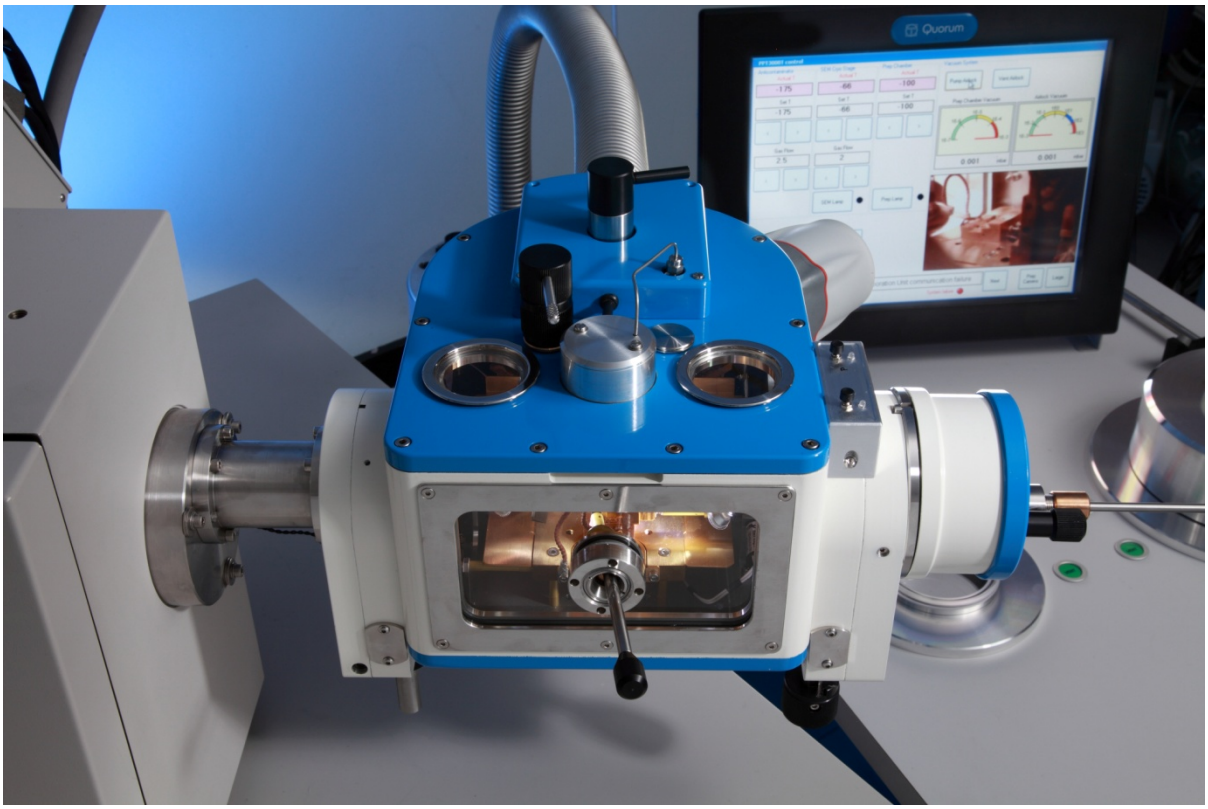
Quorum Technologies

PP3000T User manual



Contents

For technical and applications advice plus our on-line shop for spares and consumable parts visit www.quorumtech.com

Quorum Technologies Ltd is the manufacturer of a range of EM preparation equipment.

2 Acorn House
The Broyle
Ringmer
East Sussex
BN8 5NN Tel: ++44(0) 1273 815340
UK Fax: ++44(0) 1273 813439

Email : sales@quorumtech.com
Web: http:///www.quorumtech.com

For further information regarding any of the other products designed and manufactured by Quorum Technologies, contact your local representative or directly to Quorum Technologies at the address above.

- Carbon and sputter coaters
- Plasma reactor for ashing and etching
- High vacuum bench top evaporators
- Cryo transfer systems
- Critical point dryers
- Service and spares

For technical and applications advice plus our on-line shop (UK only) for spares and consumable parts visit www.quorumtech.com.

Disclaimer

The components and packages described in this document are mutually compatible and guaranteed to meet or exceed the published performance specifications. No performance guarantees, however, can be given in circumstances where these component packages are used in conjunction with equipment supplied by companies other than Quorum Technologies.

1. Health and safety

Safety is very important when using any instrumentation and this chapter should be read by all users of our equipment.
This section of the manual applies to all equipment supplied by Quorum Technologies range of products, not just the particular instrument to which the manual refers.

Included in this chapter are details on warning notations, good working practices and information on European Community (EC) legislation regarding "Control of Substances Hazardous to Health" (COSHH) and risk analysis.

1.1. Control of substances hazardous to health (COSHH)

The EC legislation regarding the "Control of Substances Hazardous to Health" requires Quorum Technologies to monitor and assess every substance entering or leaving their premises. Consequently any returned goods of whatever nature must be accompanied by a declaration form available from Quorum Technologies, reference number HSC100. Without this declaration Quorum Technologies reserves the right not to handle the substance/item. Also in accordance with EC regulations we will supply on request hazard data sheets for substances used in our instruments.

1.2. Safety policy

This section contains important information relating to all health and safety aspects of the equipment. As such it should be read, and understood, by all personnel using the instrument whether as an operator or in a service capacity.

Quorum Technologies is committed to providing a safe working environment for its employees and those that use its equipment and conducts its business responsibly, and in a manner designed to protect the health and safety of its customers, employees and the public at large. It also seeks to minimise any adverse effects that its activities may have on the environment.

Quorum Technologies regularly reviews its operations to make environmental, health and safety improvements in line with UK and European Community legislation.

Quorum Technologies cannot be held responsible for any damage, injury or consequential loss arising from the use of its equipment for any other purposes, or any unauthorised modifications made to the equipment.

All service work carried out on the equipment should only be undertaken by suitably qualified personnel. Quorum Technologies is not liable for any damage, injury or consequential loss resulting from servicing by unqualified personnel. Quorum Technologies will also not be liable for damage, injury or consequential loss resulting from incorrect operation of the instrument or modification of the instrument.

1.3. Conformity

This instrument is supplied in a form that complies with the protection requirements of the EC Electromagnetic Compatibility Directive 2004/108/EC and the essential health and safety requirements of the low voltage directive: EN61326-1 2006, EN61000-4-2, EN61000-4-3, EN61000-4-4, EN61000-4-6, EN61000-4-8. EN61000-4-11 as well as EN61010: Safety requirements for Electrical Equipment for measurement, control and laboratory use: Part 1: General requirements. Any modifications to the equipment, including electronics or cable layout may affect the compliance with these directives.

1.4. Servicing

1.4.1. Disclaimer

All service work on the equipment should be carried out by qualified personnel. Quorum Technologies cannot be liable for damage, injury or consequential loss resulting from servicing from unqualified personnel. Quorum Technologies will also not be liable for damage, injury or consequential loss resulting from incorrect operation of the instrument or modification of the instrument.

1.4.2. Operators and service engineers

A normal operator of the equipment will not be trained in or qualified for service work on the equipment and may cause a hazard to themselves or others if such work is attempted. Operators should therefore restrict themselves to the normal operation of the equipment and not by removing covers from the electronic equipment or dismantling of the instruments.

Service Engineers who are suitably trained to assess and isolate electrical, mechanical and vacuum hazards should be the only personnel who access the equipment.

1.5. Hazard signals and signs

1.5.1. Hazard signal words

The standard three hazard signal words are defined as follows:

DANGER imminently hazardous situation or unsafe practice that, if not avoided, will result in death or severe injury.

WARNING potentially hazardous situation or unsafe practice that, if not avoided, could result in death or severe injury.

CAUTION potentially hazardous situation or unsafe practice that, if not avoided, may result in minor or moderate injury or damage to equipment.

1.5.2. Hazard labels used on equipment

Several hazard symbols may be found on the equipment, they are shown in Table 1 below with their meaning:

Table 1 - Hazard warning symbols

 **Caution: risk of electric shock**	 **Caution: refer to accompanying documents**	 **Caution: Equipment is heavy. Risk of injury**

1.6. Risk analysis

1.6.1. Personal operational risks

The following is a list of tasks carried out by both the operator and service engineer where recognised risks have been observed. Listed is the personnel protection equipment (PPE) which is suggested for use for various tasks on any system.

Table 2 - Personal operational risks

Task	Carried out by: Nature of hazard	Recommended PPE
Cleaning of parts / samples with isopropanol (IPA)	Operator / service engineer. Splash hazard to eyes, drying of skin	Protective goggles, protective gloves.
Use of liquid nitrogen in sample cooling etc.	Operator / service engineer. Burn risk. Asphyxiation risk.	Thermally protective gloves and goggles should be worn. Ensure adequate ventilation.
Lifting of Heavy Items	Service engineer. Dropping on foot. Back strain.	Protective footwear. Use lifting equipment.

1.6.2. Hazardous materials

Isopropanol (IPA)

For certain service tasks isopropanol is suggested for cleaning components before use in the vacuum system. It should be noted that isopropanol is a flammable liquid and as such should not be used on hot surfaces. In addition it is recommended that protective gloves are worn when using isopropanol.

Liquid nitrogen

Only operators with experience in the safe handling of liquid nitrogen should use the equipment under these conditions. Thermally insulated gloves and goggles should be worn at all times when handling and using liquid nitrogen.

The instrument uses nitrogen and argon gas supplies for its operation; the customer is responsible for maintaining the supply to the instrument. This supply should be

regulated and kept to the lowest pressure and flow rate as is practical to minimise the effects of any leaks.

1.7. Good working practices

It is essential that good hygienic working practices are adopted at all times especially in an ultra high vacuum or cleanroom environment and are generally of the “common sense” type. Some simple good practice rules are:

- If in doubt don't.
- If in doubt ask.
- When handling solvents wear face mask, gloves, apron and work only in a well ventilated area.
- Mop up any spillages immediately.
- When handling or decanting mineral oils wear protective clothing.
- Aerosols of mineral oils, such as that produced by gas ballasting, can prove to be hazardous and an exhaust is recommended.
- Before attempting to service electrical apparatus, isolate from the mains.
- Treat all unknown substances as hazardous.
- Dispose of substances in an appropriate manner.
- Use the correct tool for the job.
- Keep a straight back and bend from the knees when lifting heavy objects.
- Wear protective clothing when using liquid nitrogen.
- Affix pressurised gas cylinders firmly to walls or racks. Use the correct regulating valves on gas cylinders and always transport cylinders using the appropriate specialist trolley.
- Obey safety regulations regarding lifts, hoists and machine tools.
- Always make sure you understand a procedure well before attempting it for the first time.

1.8. Quorum PP3000T specific safety hazards

The following Safety Hazards are specific to the Quorum PP3000T Cryo Transfer System.

1.8.1. Gases

Nitrogen and argon gas supplies

This instrument uses nitrogen and argon gas supplies for operation; the customer is responsible for maintaining the supply to the instrument. This supply should be regulated and kept to the lowest pressure and flow rate as is practical to minimise the effects of any leaks.

Extreme care to be used when filling the slusher assembly with liquid nitrogen, follow your company’s standard Health and Safety instruction when using this liquid gas.

1.9. Scope

This manual is intended for all users of the PP3000T Cryo System manufactured by Quorum Technologies and provides information on the operation and maintenance of the instrument.

Please note that the servicing and maintenance procedures should only be carried out by qualified service personnel and it is essential that all users should read the Health and Safety section of this manual.

1.10. Return of goods

If goods are to be returned to Quorum Technologies for repair or servicing the customer should contact their local distributor or the factory direct before shipment. A "Returns Authorisation Number" should be obtained in advance of any shipment. This number is to be clearly marked on the outside of the shipment.

All returned goods are to be accompanied by a completed "Returned Goods Health and Safety Clearance" form HSC-100 attached to the outside of the package (to be accessible without opening the package) and a copy of the forms should be faxed in advance to the factory.

A copy of this form can be found at the end of the manual or requested directly from Quorum Technologies, the details can be found on page two of this document.

1.11. Returns procedure

1.11.1. Warranty claim

Electronic and basic servicing capabilities exist at most in-country appointed agents, however all components are sold with a return to factory warranty (unless otherwise stated) which covers failure during the first 12 months after delivery. This is extended by a further 2 years if the warranty form is returned to Quorum Technologies.

Returns must be sent carriage paid, Quorum Technologies will cover the return carriage costs. This covers defects which arise as a result of a failure in design or manufacturing. It is a condition of warranty that equipment must be used in accordance with the manufacturers' instructions and not have been subjected to misuse. This warranty does not cover consumable items such as sputter coating targets and carbon evaporation material. To make a claim under the terms of this warranty provision contact the Customer Service Department of your local Quorum Technologies representative in the first instance.

1.11.2. Chargeable repairs

Always contact your local Quorum Technologies representative in the first instance. They will be pleased to assist you and will be able to provide an estimate of repair costs; many offer local repair facilities.
For routine repairs, where down-time is not critical, the target standard return time at Quorum Technologies is 20 working days.

1.11.3. Packaging and carriage

All goods shipped to the factory must be sealed inside a clean plastic bag and packed in a suitable carton. If the original packaging is not available, Quorum Technologies should be contacted for advice. Quorum Technologies will not be responsible for damage resulting from inadequate returns packaging or contamination of delicate structures by stray particles under any circumstances. All non-warranty goods returned to the factory must be sent carriage pre-paid, (Free Domicile). They will be returned carriage forward (Ex-Works).

2. Introduction

The PP3000T Cryotransfer system allows users to prepare, load, process and transfer cryo specimens into an SEM. The specimen may then be imaged at cryogenic temperatures in the SEM chamber.

2.1. Basic overview of system

2.1.1. Stage bias voltage

Please note that the standard cryo-SEM sample stage supplied with the Quorum Technologies Ltd cryo-SEM system is not suitable for use with stage bias.

We have become aware of instances where the Quorum Technologies Ltd, cryo-SEM instrument has been used in conjunction with sample stage high voltage bias, resulting in considerable damage to the control electronics of the cryo instrument.

Use of sample stage bias with the cryo-SEM system will void all warranty. Damage resulting from use of stage bias with the cryo-SEM and standard stage will be extensive and costly to repair.

The system can be broken down into five main units:

- SEM cold stage and anti-contaminator
- Column mounted preparation chamber
- PrepDek® specimen preparation desk
- Turbo pumping stack
- CHE3000 cold gas cooling system

2.2. SEM cold stage and anti-contaminator

2.2.1. SEM cold stage (Figure 1)

The SEM cold stage has two cartridge heaters and a Pt100 temperature sensor fitted. It sits on high efficiency thermal mounts and is cooled by cold nitrogen gas supplied by the off-column cooling system. The temperature is controlled by a combination of gas flow and heating between about -193°C and +50°C. If required the specimen may be sublimated in the microscope whilst simultaneously imaging to establish optimal thermal and temporal conditions which can then be used in the preparation chamber. The cold stage does not, normally, limit the tilt or translation of the microscope stage. An optional rotate stage allows full 360° rotation of the specimen. A STEM version of the cold stage is also available.

When not in use and where space in the SEM chamber allows, the cold stage may be parked in the supplied retainer inside the microscope chamber and does not need to be removed for normal microscope operation. The anti-contaminator may be left in place and used in addition to the standard microscope cold trap or swung to one side. The cold stage is mounted on the standard SEM stage using an adapter plate. In most installations it will be necessary to remove the normal SEM specimen holding device in order to fit the cryo stage.

Figure 1 - Example SEM cold stage

2.2.2. SEM anti-contaminator (Figure 2)

The SEM anti-contaminator is mounted on a ball-jointed support and can be swung out of the way when not in use. The anti-contaminator is fitted with a Pt100 temperature sensor. The temperature may be varied between -193°C and ambient and allows rapid warm up of the system. The shape of the anti-contaminator is tailored to suit the SEM to which it is fitted.

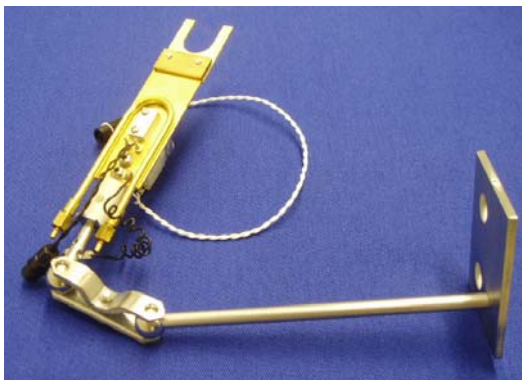


Figure 2 – Example SEM anti-contaminator

2.2.3. SEM chamber illumination

To ensure good visibility when inserting a specimen, the SEM chamber is fitted with high intensity LED illumination. The LEDs are located close to the transfer rod axis and give excellent visibility when loading the specimen shuttle.

2.2.4. SEM chamber CCD camera

Where no chamberscope is available and space allows, a CCD camera may also be fitted to the SEM chamber. The image is displayed on the touch screen and can be enlarged to fill the screen when transferring.

2.3. Column mounted preparation chamber (Figure 3)

The preparation chamber has been designed to maximise visibility of the specimen during specimen preparation and transfer. Large cold surfaces provide excellent trapping of sublimed water vapour as well as cryo pumping the chamber. The integral non-boiling dewar cools the cold stage, cold shields and specimen manipulators. Vibration from boiling liquid nitrogen which could be transferred to the SEM are eliminated. A multi-range vacuum gauge is mounted directly on the chamber giving an accurate reading of the vacuum level close to the specimen.

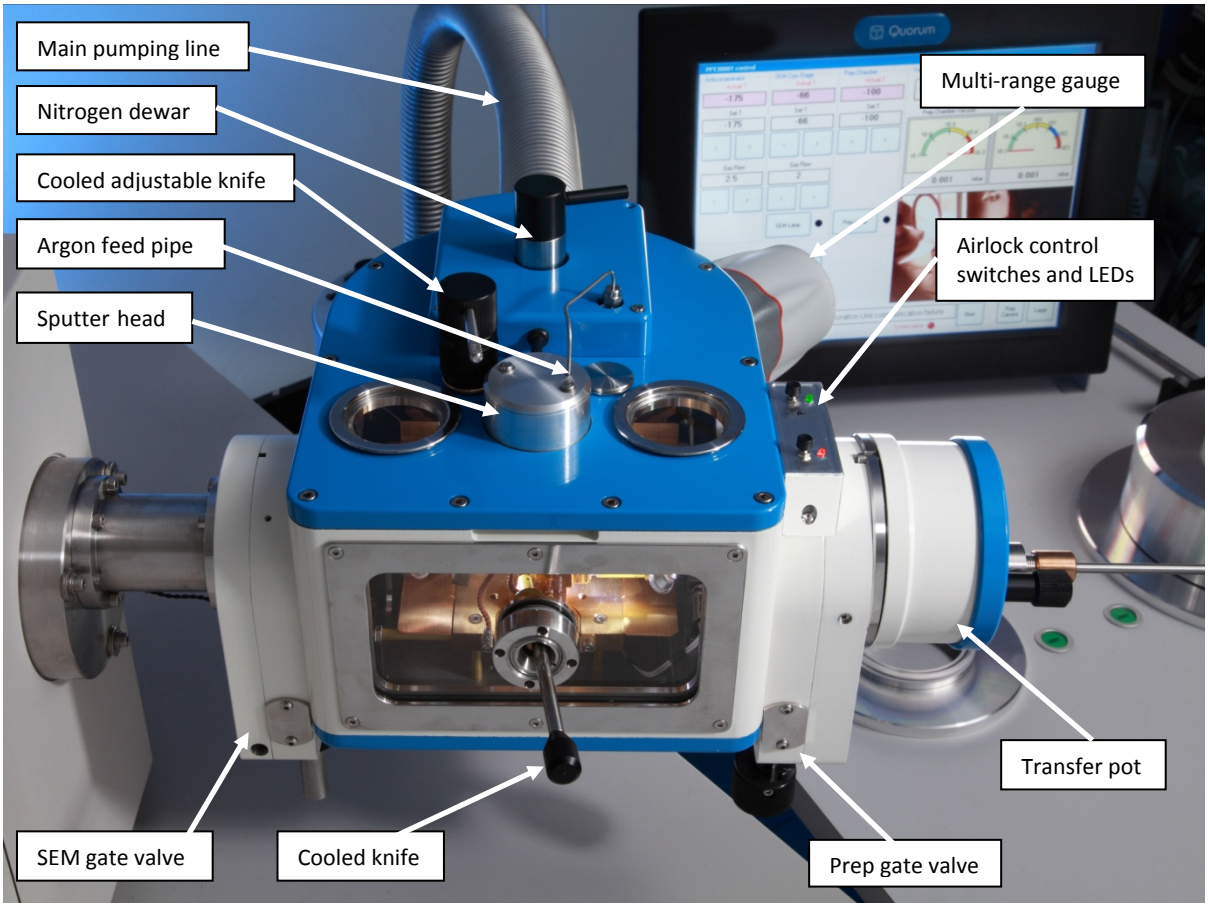


Figure 3 - Preparation chamber

Within the preparation chamber are:

2.3.1. Cold stage

The cold stage fitted to the preparation chamber has twin cartridge heaters and a Pt100 temperature sensor. It sits on a cold block and is separated from this

block by a thermal break. This allows the cold stage temperature to be varied without boiling off a considerable amount of liquid nitrogen. The cold stage temperature can be varied between about -185°C and +50°C with a stability of 1°C and is controlled by one channel of the three channel dedicated temperature control system. The low thermal mass of the cold stage allows rapid temperature stabilisation.

The main cold block is connected to the liquid nitrogen dewar via copper braids which allow for any movement due to thermal contraction.

2.3.2. Cold trap

The cold trap is mounted directly onto the cold block and sits close to liquid nitrogen temperature. It is made of gold-plated copper for optimal thermal performance.

2.3.3. Illumination

Four focussed high intensity white LEDs illuminate the specimen. These are adjustable and easily removed for cleaning. Shadowing of the specimen by fracturing tools is minimised.

2.3.4. CCD camera (see Figure 4)

A small CCD camera looks through one of the side windows and is focussed on the specimen shuttle and bayonet fitting area. The image from this camera is displayed on the touch screen and can be enlarged to fill the screen making viewing of fracturing and bayonet engagement very easy. The window is protected from sputtered material by a solenoid operated shutter.

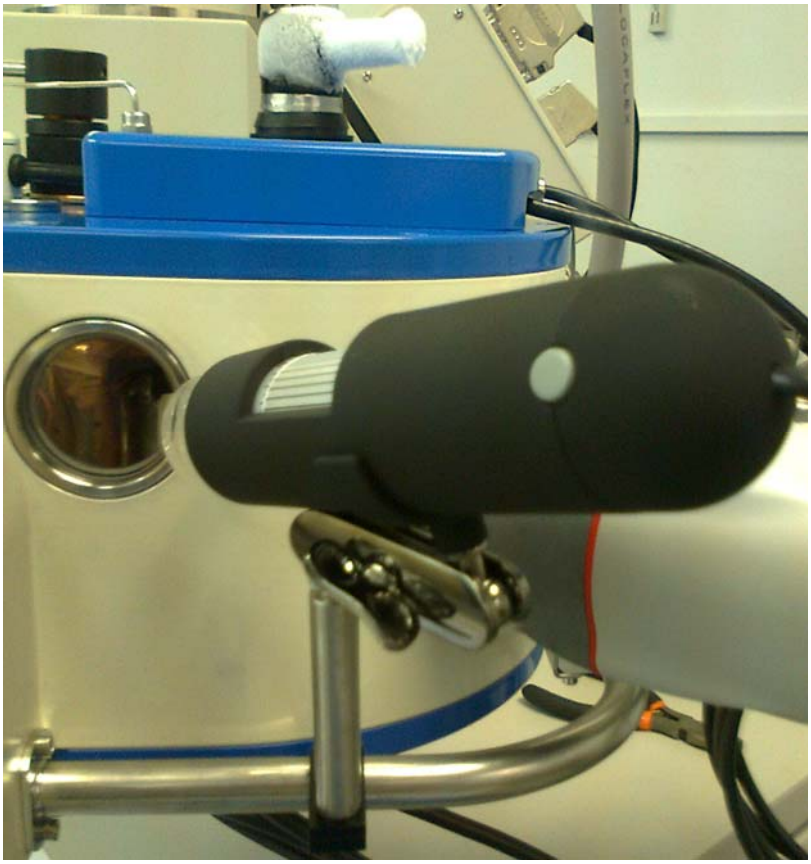


Figure 4 - CCD camera on preparation chamber

2.3.5. Sputtering and carbon coating heads

The standard coating option supplied is metal sputtering. A carbon head is available as an option for those wishing to do EDS analysis. The whole sputtering process is automated from argon gas introduction to striking of the plasma and current control. Many different metal targets are available for the sputter head. Platinum is supplied as standard. Iridium is the recommended option for the finest grain size.

2.3.6. Film thickness monitor

An optional film thickness monitor mounted behind the cold block enables actual coating thickness to be measured if required.

2.3.7. Liquid nitrogen dewar

The large integral liquid nitrogen dewar with high thermal efficiency and low boil off cools the cold block and stage. The inner surface of the dewar is coated to prevent boiling and is made of aluminium to reduce weight. The dewar is mounted from the top lid and is located by plastic isolators to prevent vibration.

2.3.8. Gate valves

Two gate valves isolate the prep chamber from the SEM and transfer airlock. These valves are solenoid interlocked to prevent opening when the vacuum levels are incorrect. A bi-colour LED shows the valve status. The SEM valve provides interlocking signals to the SEM and where the necessary signals are made available by the SEM manufacturer can be disabled by the microscope.

2.3.9. Cryo transfer device (see Figure 6)

The specimen is transferred cold and under vacuum from the PrepDek® slusher to the airlock using a dedicated transfer device. This device has a sealed chamber which is pumped out on the slusher and then closed. The specimen shuttle (Figure 5) latches onto the thermally isolated transfer rod via a bayonet fitting which engages and disengages by rotating through 90°. A pointer on the outer end of the transfer rod indicates the orientation of the bayonet.

A window in the back of this device allows observation of the specimen as it is transferred into the preparation chamber or onto the SEM cold stage.

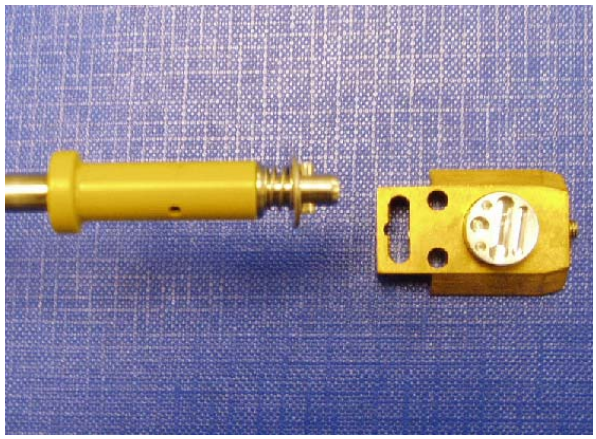


Figure 5 - Specimen dovetail and bayonet fitting

Figure 6 – Cryo transfer device front view

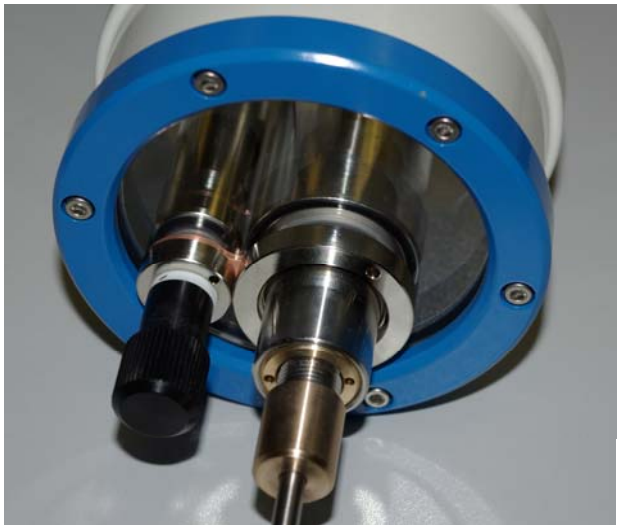


Figure 7 – Cryo transfer device rear view

2.4. PrepDek® specimen preparation desk

The workstation, trademarked PrepDek®, houses the dual slusher pots for specimen preparation and manipulation, the control electronics and the panel PC. Each of the slusher pots may be evacuated and vented independently. The larger of the two pots is fitted with a manipulator for loading pre-frozen specimens from high pressure freezers, plunge freezers, cryo-microtomes or those prepared off site. A bright flexible LED lamp provides illumination of the pots. Each pot can have a dark coloured disk fitted in the bottom to give high contrast when loading under liquid nitrogen.

A storage tube is provided for the transfer device which enables it to be stored under vacuum keeping it clean and moisture free.

Figure 8 - PrepDek slusher pots

Figure 9 - Transfer device in storage tube

2.4.1. Control electronics

All the control electronics are mounted in a sealed case fitted below the PrepDek®. Connections are made from below to prevent any ingress of water or liquid nitrogen. The power supplies and valving are all controlled from a touch screen computer mounted on an adjustable arm on top of the workstation.

Control of stage and anti-contaminator temperatures and cooling gas flow is via a dedicated multi-channel temperature control system. Valving and sensing is controlled by another digital I/O card and all gauge readings are made via the vacuum control board. The system is designed to be modular and simple to diagnose should any fault occur. The main power unit supplies the sputter high voltage, the evaporation low voltage-high current and the system 24V. Each unit is stand alone and communicates via RS485 protocol with the main control PC.

2.5. Turbo pumping stack

The system is evacuated by a turbo pumping stack. This stack has an integral buffer tank which means the backing pump only runs a few minutes in each hour. A Pirani gauge monitors the buffer tank pressure and triggers the control PC when the tank needs to be pumped. Another Pirani gauge monitors the backing pressure and is also used for system and airlock pump monitoring. The pump stack has a heavy anti-vibration base block to prevent transfer of vibration to the SEM chamber. Connection from the turbo to the preparation chamber is by a stainless steel vacuum bellows. All valving is integrated into the pump stack and is controlled via the PC.

Caution: The pump stack is heavy. Use the supplied strap if it needs to be moved. This requires two people.

2.6. CHE3000 cold gas cooling system

An off-column, dual circuit gas cooling system, CHE3000 provides the cooling for the SEM cold stage and anti-contaminator. Each circuit is controlled independently allowing a large temperature differential to be set between the specimen and anti-contaminator for optimal cryo trapping. The twin heat exchanger has integrated drying stacks to remove any water in the supplied nitrogen gas. The transfer tubes are vacuum isolated and a sorb pump maintains this vacuum so the vacuum isolation only needs occasional pumping. The CHE3000 is fitted as standard to PP3000T and has a number of significant advantages:

- Although column mounted dewars fitted to many previous Quorum cryo systems are very efficient because the volume of the dewar is actively pumped by the SEM pumping system, their capacity is relatively small and therefore they only offer a limited hold time between refills.

- This limited hold time can be problematic for some users and limiting for some cryo applications (e.g. unattended operation). It also adds weight and surface area which can transfer vibration to the microscope column.

- On some “heavily loaded” SEMs there is often a lack of clearance to other accessories to enable the fitting of an on-column dewar; there may be a free SEM port, but no space above for the dewar.

- The CHE3000 has a 12 litre liquid nitrogen capacity allowing run times between refills of over 16 hours.

Typically the CHE3000 needs to be located behind the SEM and is connected to the SEM through tubes. Access is needed to the dewar for filling.

The system has been designed with ease of use in mind. The entire system is controlled by a touch screen PC. This is mounted on a monitor arm attached to the PrepDek® allowing it to be positioned to suit the user. Most functions have been automated and the user can save personal settings for specimen coating and sublimation as “recipes” for future use. The PrepDek® workstation allows the user to work with pre-frozen specimens as well as to freeze their own. PrepDek® designed as an expandable system where additional features will be added in the future.

Holders are available for virtually any type of specimen from liquids to rocks. Special holders can also be developed to suit the users’ specimens if required.

Once the system has been cooled down and reached base temperature, it will run all day without the need to refill the CHE3000 dewar. The preparation chamber dewar hold time will depend on the number and temperature of sublimation cycles.

Continuing the ease of use philosophy, disassembly and cleaning of the chamber is simple with a minimum of tools or dismantling required. Windows are piston sealed and can be pulled out for cleaning. The top and base are easily removable.

3. Software

On start up the system loads the control software and displays the main control window. This window is split into logical functional areas.

- Top data bar
- Temperature monitoring and control
- Gas flow monitoring and control
- Vacuum monitoring and control
- Process control
- Imaging control
- Messaging areas

Parameters on the screen which can be changed by the user are surrounded by a dark outline; parameters which are read only are surrounded by a grey frame.

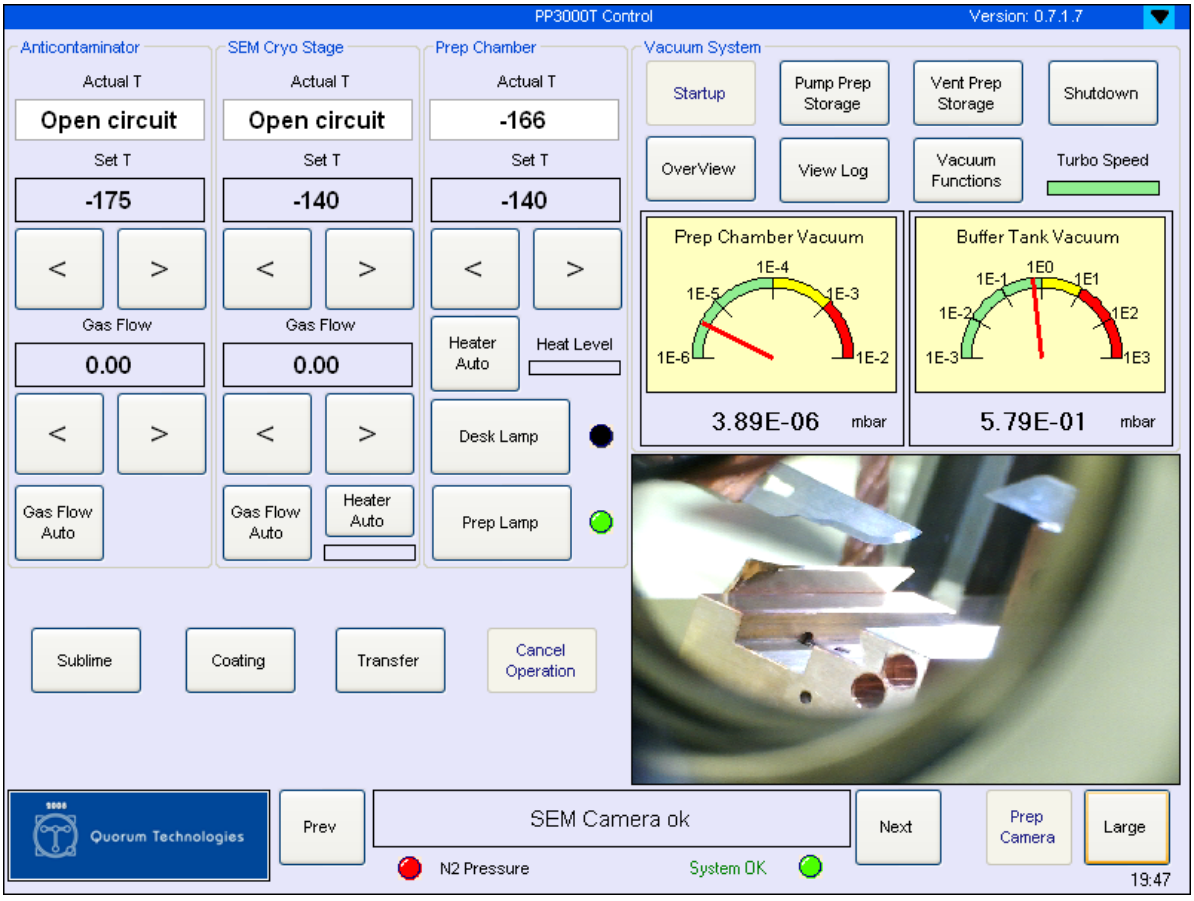


Figure 10 - Main start up screen

3.1. Top data bar

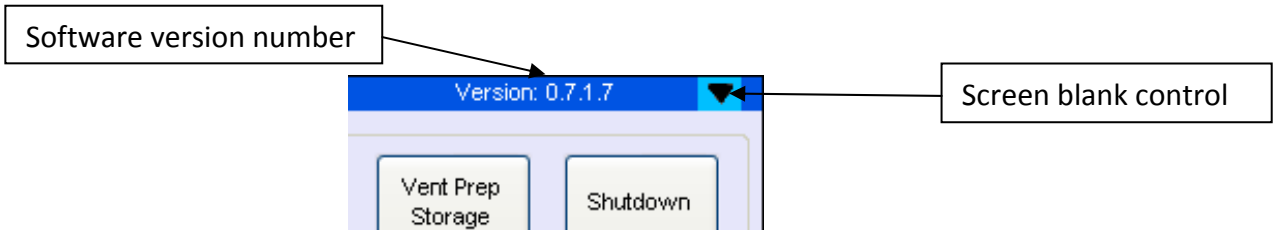


Figure 11 - Blanking button and software version number

The screen may be blanked at any time by touching the down arrow icon in the top right hand corner of the screen. Touching anywhere on the blanked screen will restore the control interface.

The software version number is displayed next to the blanking icon.

3.2. Temperature monitoring and control

The temperature of the SEM anti-contaminator, SEM cold stage and preparation chamber cold stage are continuously monitored and displayed.

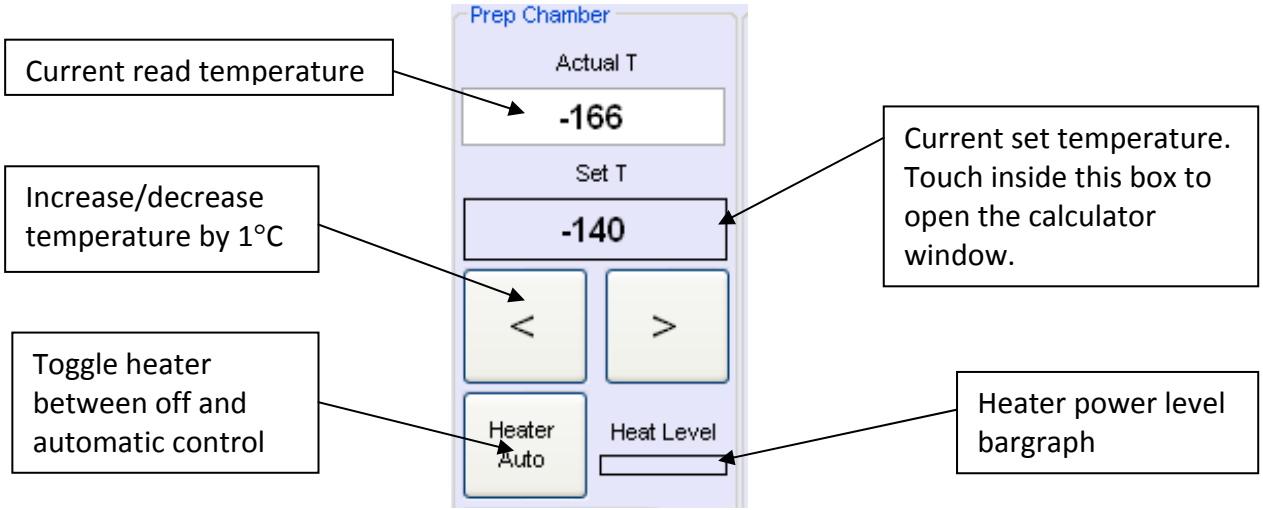


Figure 12 - Temperature control area

The temperature controls work in the same way for each of the three areas (SEM cold stage, SEM anti-contaminator, preparation chamber cold stage).

The current temperature is displayed at the top of the box. This is read only. If a sensor is not connected or a wire broken the current temperature will read “Open circuit”. Below this the current set temperature is displayed. This can be changed by using either the up down arrow buttons to make small increments or decrements or, if a larger change is required, by touching the set temperature display area. This will call up a calculator window (Figure 13) where the desired temperature may be entered.

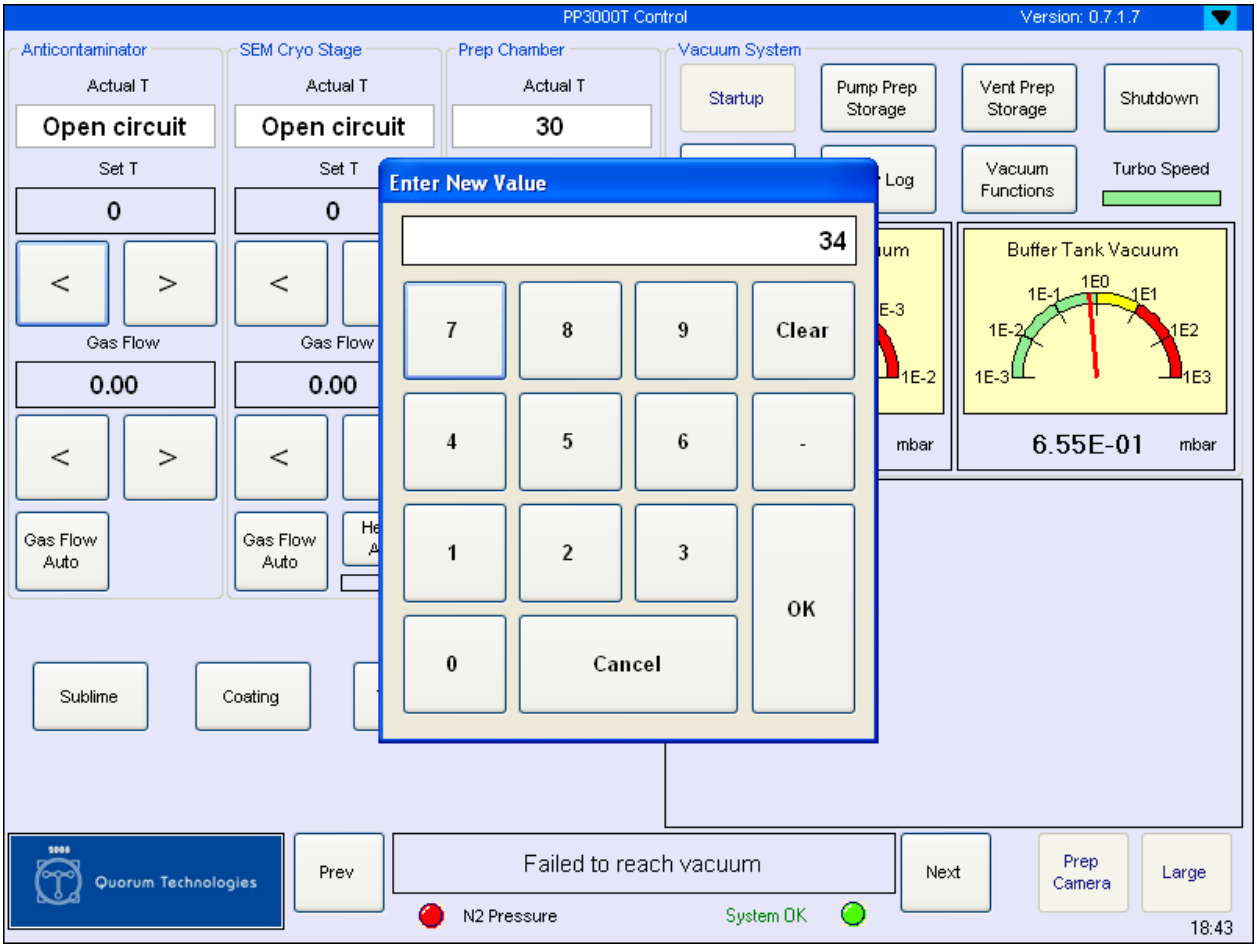


Figure 13 - Changing the set temperature

The minus sign (-) may be pressed at any time when entering the new set temperature. If a data entry error is made it can be corrected by touching the “Clear” button. Touching “OK” will transfer the new value to the set temperature window. Touching the “Cancel” button will exit with no change being made to the set temperature.

3.3. Gas flow monitoring and control

Two independent gas flow circuits control the flow of cold nitrogen gas through the heat exchangers of the SEM anti-contaminator and SEM cold stage. The flow of gas is regulated by a proportional valve and the flow measured by a mass flow meter. The flows are displayed below their respective temperature displays (Figure 14).

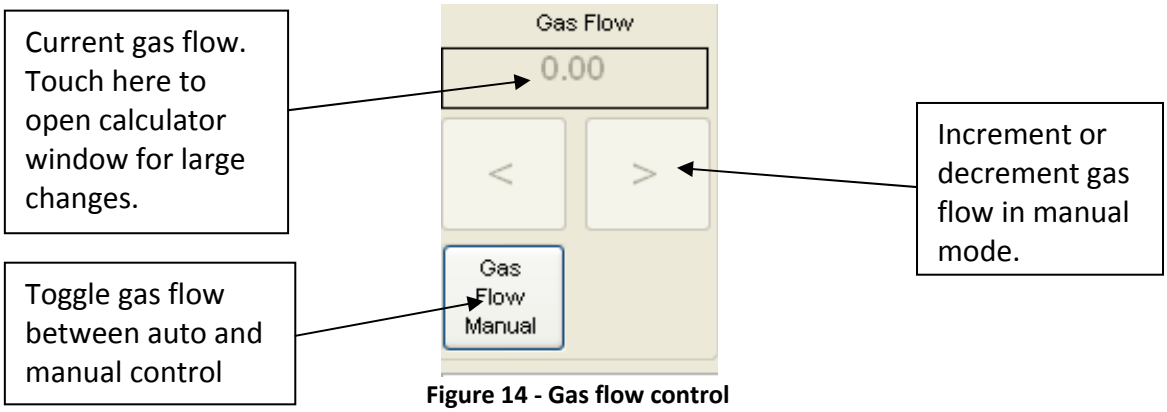


Figure 14 - Gas flow control

The temperatures may be controlled by gas flow alone or by a combination of gas flow and heating depending on the mode selected. To minimise the gas consumption, it is usual to control with gas flow only when not subliming. The system defaults to manual gas control.

3.4. Vacuum monitoring and control

The top right hand area of the touch screen displays the vacuum monitoring and control functions.

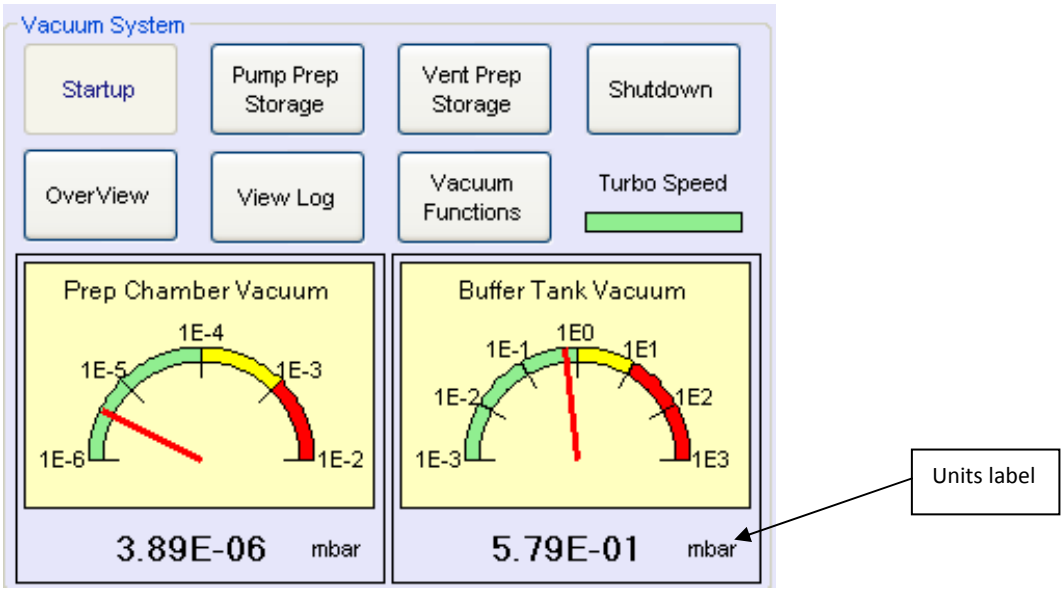


Figure 15 - Vacuum monitoring and control area

3.4.1. Vacuum monitoring

Vacuum levels in different parts of the system are displayed within the “Vacuum System” frame.

The left hand meter (Figure 15) is for the “Prep Chamber Vacuum” and is read from a wide range gauge mounted directly on the preparation chamber. This gauge can read from atmospheric pressure down to about $1e^{-8}$ mbar. The units may be displayed in either Pascal or millibars. The units are changed by touching the units label.

The right hand gauge displays one of the two Pirani gauges fitted to the system, depending on the function which is running at the time. The caption above the gauge changes to show which part of the vacuum system is being displayed. Normally, this is the turbo buffer tank vacuum. When the airlock is being pumped out the “Airlock Vacuum” gauge is displayed. When the vacuum isolated line is being pumped “Isolated vacuum line” is displayed. The buffer tank vacuum is monitored above the gauge display. Both analogue and digital displays are shown. The analogue display makes checking vacuum levels easy from a distance as the needle only needs to be in the green band.

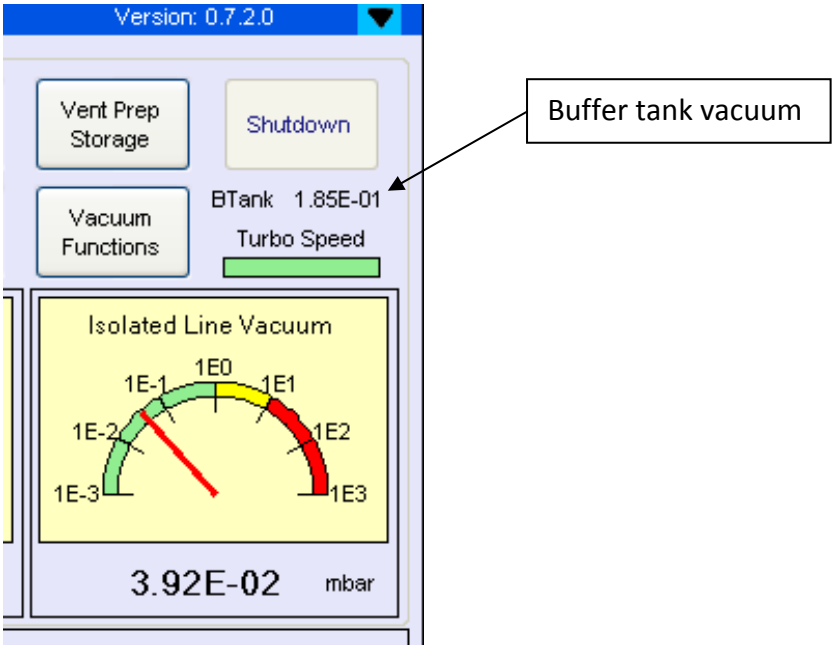


Figure 16 - Pumping the vacuum isolated line

3.4.2. Vacuum control

Within the “Vacuum System” frame are several control buttons:

- Startup
- Shutdown
- Pump Prep Storage
- Vent Prep Storage
- Vacuum Functions
- Overview
- View Log

as well as the turbo speed indicator. The speed indicator bar will change colour from red (<80%) through orange (<90%) to green (>90%) depending on the turbo speed.

3.4.2.1. "Startup"

The vacuum system is automated and will begin its start up procedure when this button is touched. On touching the "Startup" button, a small options window opens. Here the user can decide if they want to run a fully automated cryo session, which guides the user though the full start up and loading procedure, or to use the PP3000T in manual mode. Should the user want to log the pump down and cool down the tick box labelled "Log vacuum and temperature values" should be touched.

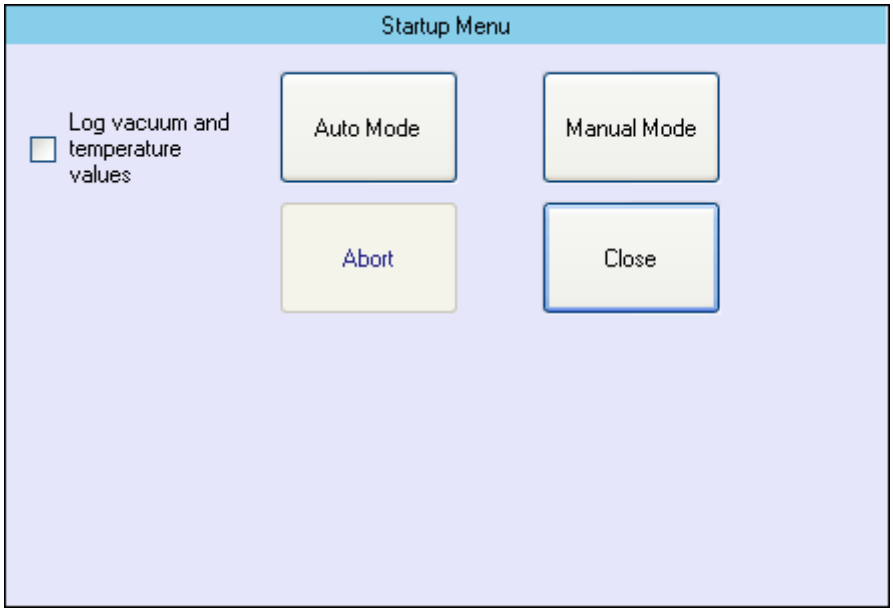


Figure 17 – Start up pop up

3.4.2.2. "Shutdown"

Pressing this button starts the automated shut down procedure.

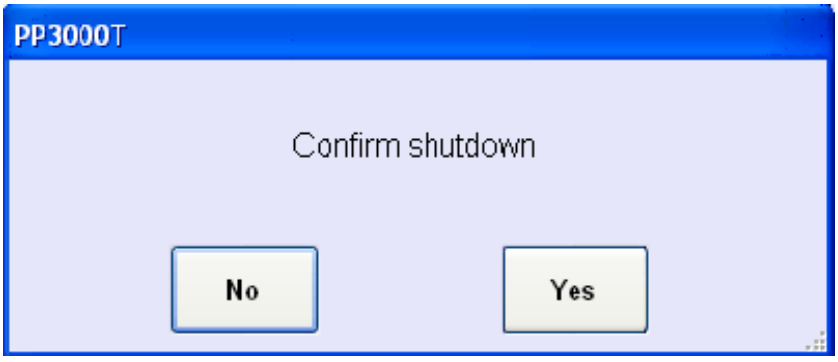


Figure 18 - Shutdown initial screen

Figure 18 shows the shutdown confirmation screen.

3.4.2.3. "Pump Prep Storage"

This button starts the pump out of the transfer device storage tube.
NB: The transfer device should be in the tube before starting to pump.

3.4.2.1. "Vent Prep Storage"

The transfer device is normally kept under vacuum in the storage tube. To vent this tube and remove the transfer device, simply touch this button.

3.4.2.2. "Vacuum Functions"

3.4.2.2.1. "Pump Vacuum Isolated Line"

This button starts the pump out of the CHE3000 vacuum isolated line. NB: The quick release valve should be fitted to the top of the CHE3000 before touching this button.

3.4.2.2.2. "Vent Vacuum Isolated Line"

Where the CHE3000 vacuum isolated lines have been pumped and the sorb pump regenerated, pressing this button will vent the pumping line and it may then be disconnected from the vacuum isolated line.

NB: The quick release valve on the top of the CHE3000 must be closed first.

3.4.2.2.3. "System vent"

This button will vent the entire system provided it is not cold.

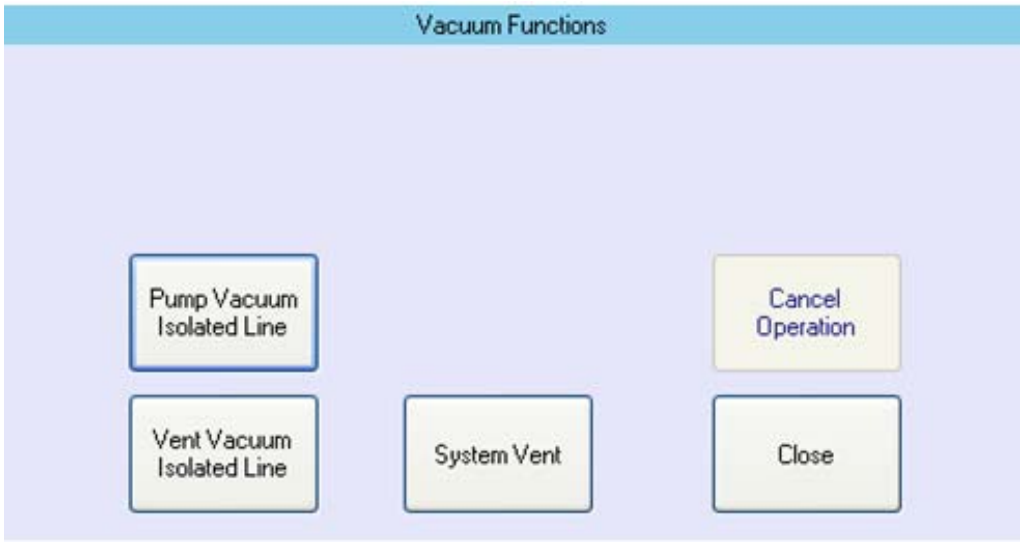


Figure 19 - Vacuum functions window

3.4.2.3. Overview

This button calls up a graphical representation of the system showing the status of all valves, switches etc. It is read only.

3.4.2.4. View Log

This button will call up the graphs of pump down and cool down.

3.4.3. Logging the system parameters

If the “Log vacuum and temperature values” tick box is selected on “Startup” the system will store data for temperatures and vacuum in a log file.

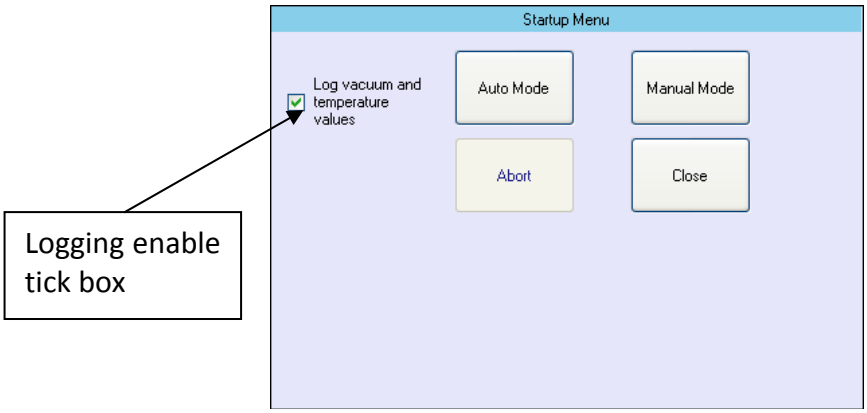


Figure 20 - Activating the data logger

The data can be displayed graphically by touching the “View Log” button. This is a toggle button, so the display may be masked by touching again.

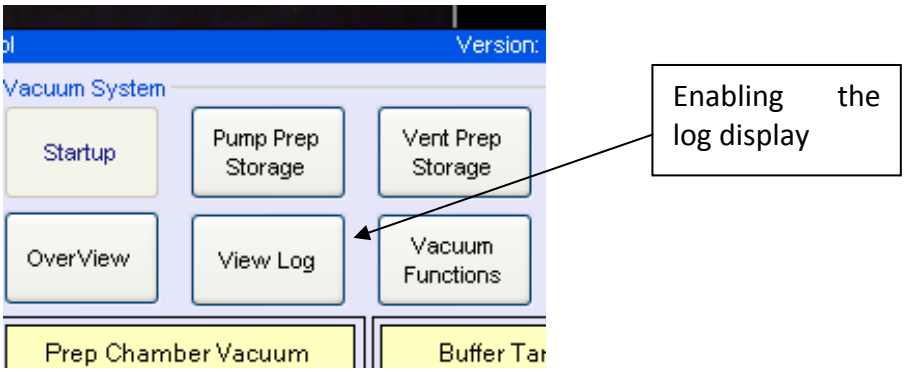


Figure 21 - Enabling the log display

Individual parameters are displayed by selecting the appropriate button below the graph. The data is not live but can be refreshed by touching the “Update” button.

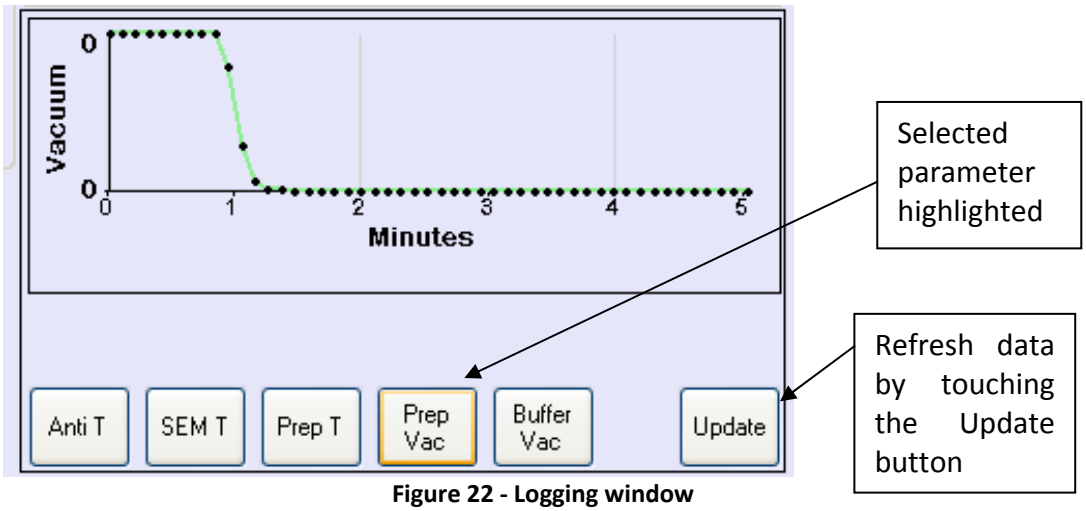


Figure 22 - Logging window

3.5. Process control

Once a specimen has been transferred to the preparation chamber it may be sublimed, coated and then transferred into the SEM chamber. These processes are activated by the process control buttons (Figure 23).

The buttons are laid out in the order they will most probably be used across the screen. The “Evaporation” button only appears if the optional carbon evaporation board is fitted.

Figure 23 - Process control buttons - no evaporation board fitted

3.6. Sublimation

Touching the “Sublime” button calls up the sublimation control screen. Sublimation is done via “recipes” which the user can define and save for future use. Up to three commonly used “recipes” can be assigned to quick access buttons for instant recall. The profile of the defined sublimation curve is shown graphically in the centre of the frame with an approximation of the warming and cooling ramps. The profile can be edited or a new one defined using the “New” or “Edit” buttons.
Sublimation can be carried out in the preparation chamber or in the SEM chamber. The stage (SEM or Prep) which is to be used for sublimation is selected by a radio button.

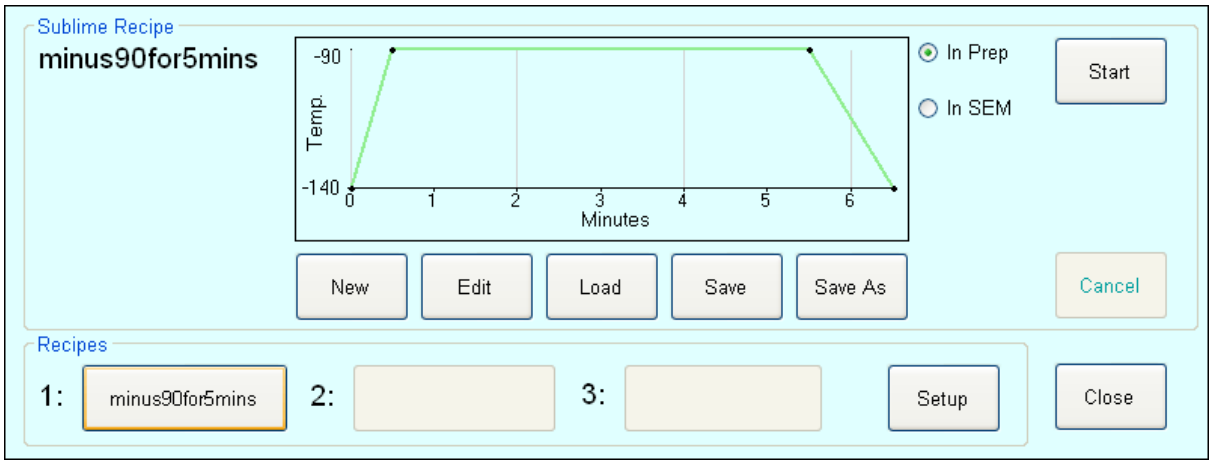


Figure 24 - Main sublimation screen

3.6.1.1. Defining a new sublimation recipe

When the “New” button is touched a pop up window appears which is very similar to the “Edit” screen but there will be no temperatures or times defined. The window is described below in 3.6.1.2.

3.6.1.2. Editing an existing sublimation recipe

Touching the “Edit” button opens a pop-up window (Figure 25) which displays the shape of the current defined sublimation curve and buttons which allow the values to be edited, new steps to be added at any point in the overall curve, or to delete certain points. Touching the up and down arrow buttons scrolls through the defined steps.

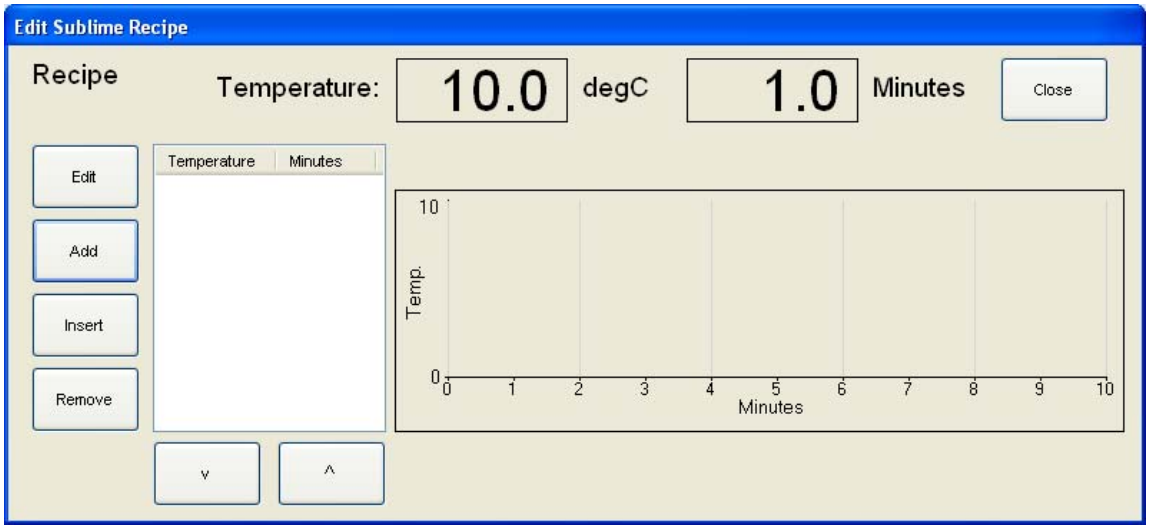


Figure 25 - Editing the sublime recipe

The “Edit” button allows the user to change the temperature or time of the currently selected point.
The “Add” button will add another temperature and hold time to the end of the list.
The “Insert” button will add another temperature and hold time above the currently selected point.
The “Remove” button will delete the currently selected temperature and hold time.

As the curve is edited the graphic will change to reflect the edits.

Touching the “Close” button will transfer the updated curve to the sublime window and close the editing window.

3.6.1.3. Assigning a recipe to a quick access button

In the “Recipe” frame are three quick access buttons (labelled 1, 2 and 3) and a “Setup” button. When “Setup” is touched another pop up window appears with the available recipes displayed. The user touches the file name of the required recipe and touches the “OK” button. The selected recipe will be assigned to the quick access button. Touching “Cancel” will close the window with no changes.

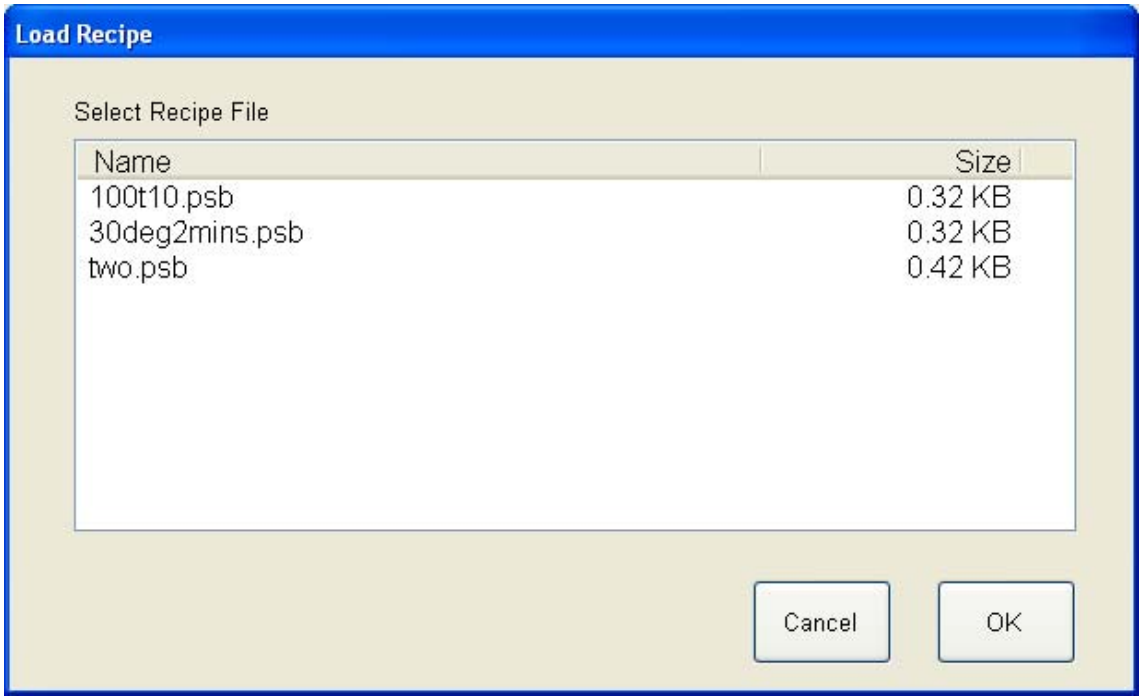


Figure 26 - Loading a stored recipe

3.6.1.4. Saving a defined recipe

If a recipe has been defined which will be used again it can be saved to disk (Figure 27) for recalling at a later date. If this is a change to an existing recipe touching the “Save” button will store the recipe under the same file name and overwrite the current values. Touching the “Save as” button will open another window where the recipe may be saved under a new file name using the touch screen keyboard.

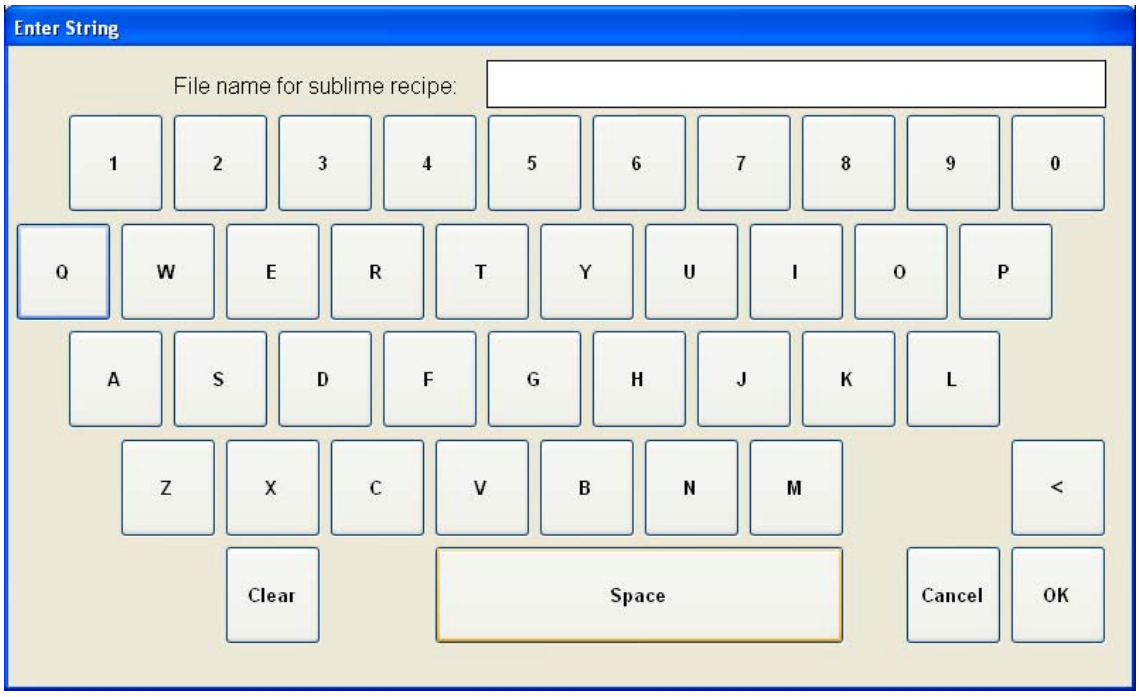


Figure 27 - Saving a new recipe

Once the required recipe has been loaded the user just has to press “Start” for the sublimation to proceed automatically. A red “snail trail” line shows the actual sublimation profile as compared to the set profile. Sublimation can be aborted at any time by touching the “Abort” button. Once sublimation is completed the window can be closed using the “Close” button.

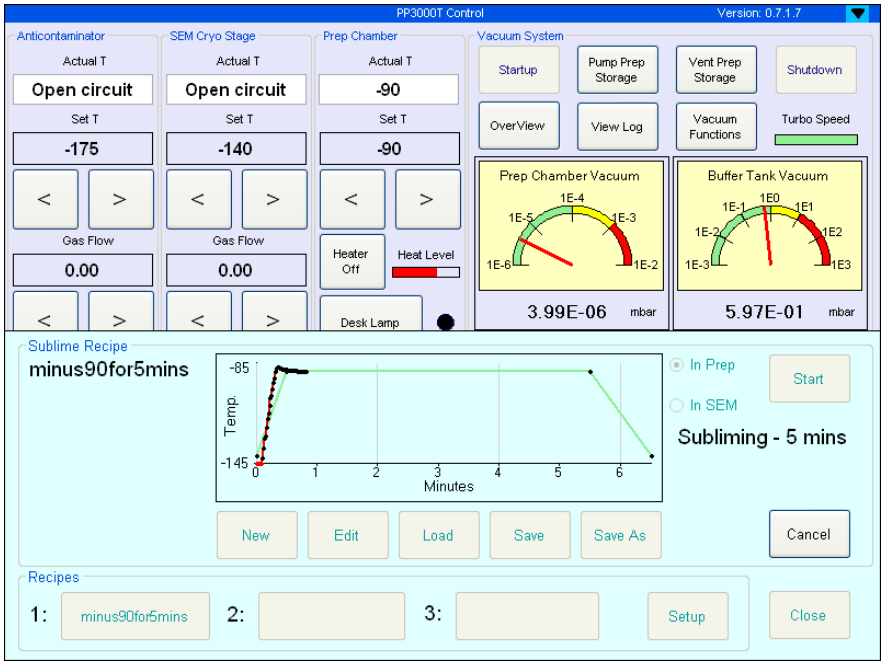


Figure 28 - Sublimation started

3.7. Coating the specimen

Once the specimen has been sufficiently sublimated it can then be coated with a fine layer of metal to render it conductive. Once again, coating is done using recipes in much the same way as for sublimation. Touching the “Sputter“ button opens the sputter coating window (Figure 29).

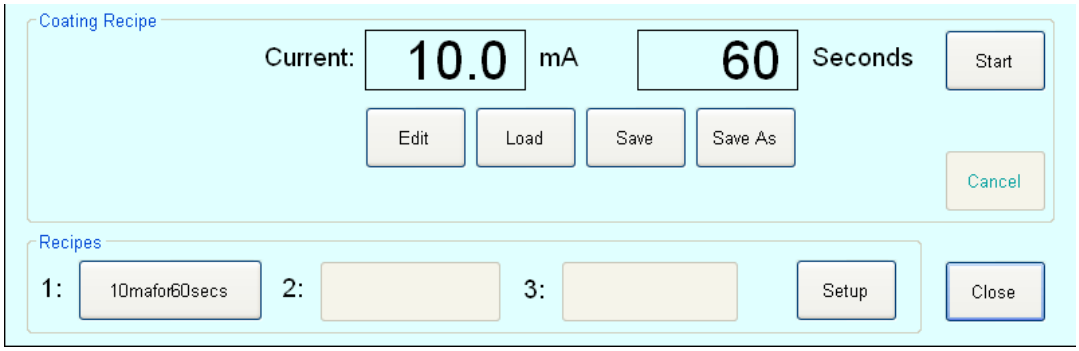


Figure 29 - Coating recipe screen

Editing, saving and defining recipes in this window function in the same way as for the sublimation menu. Only one set of time and current may be defined.

Once a recipe is defined the user just has to touch the “Start” button for the sputter coating to run under fully automated control.

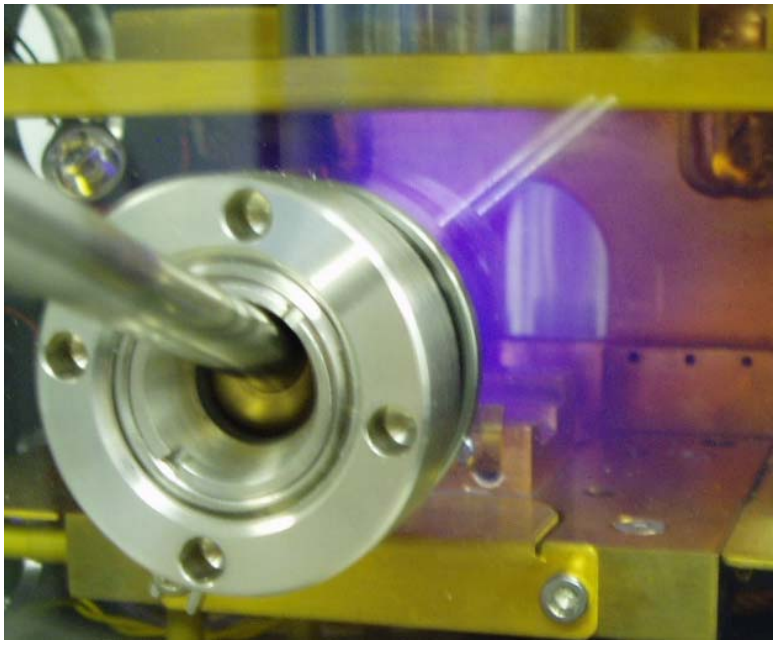


Figure 30 - Sputter plasma under automatic control

3.7.1. Using the optional film thickness monitor

If the optional film thickness monitor is fitted the sputtering may be controlled by the monitor. The FTM button should be touched to access the FTM control panel.

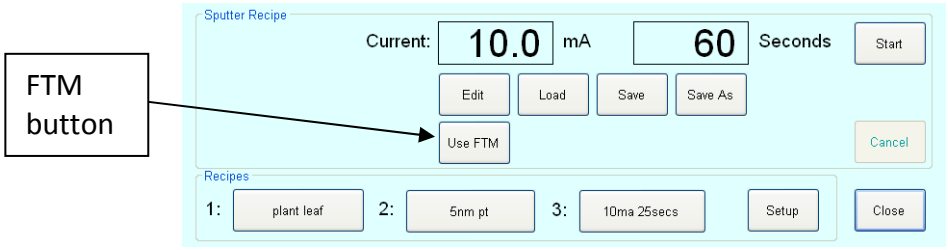


Figure 31 - FTM button

The FTM menu will then appear.

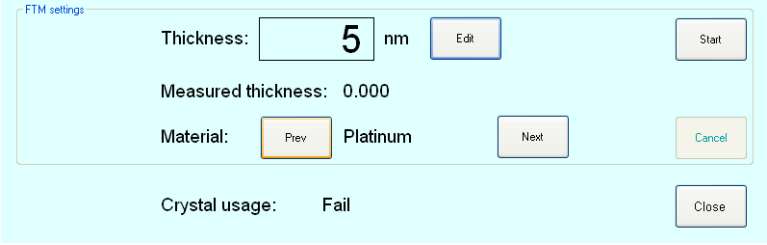


Figure 32 - FTM menu

The default target material will be displayed as well as the measured thickness. To change the target material touch either the “NEXT” or “PREV” buttons to cycle

through available materials. If the Crystal usage shows “Fail” either the cable is unplugged or the crystal has been used to exhaustion and will need to be replaced. Touching the edit button will allow the desired thickness to be entered. To start the coating, touch “START”. The measured thickness will be zeroed and the sputter coating will run automatically and stop once the set thickness is reached.

3.7.2. Replacing the FTM crystal

The FTM is accessed through the front window.

- Vent the system
- Unscrew the 6 countersunk cap screws which hold the front bezel and glass in place
- Loosen the cap screw which holds the front knife braid in place and remove the window
- Wearing a pair of gloves, lift the FTM body from the spring clip
- Unplug the miniature BNC connector
- Unscrew the top retaining ring on the FTM body
- Remove the crystal
- Insert the new crystal
- Clean any sputtered material from the retaining ring and replace
- Reconnect the miniature BNC connector
- Replace the FTM body in the spring clip
- Check the FTM control page to check the crystal usage is now zeroed
- Replace the front window, knife braid and front bezel
- Re-pump the system

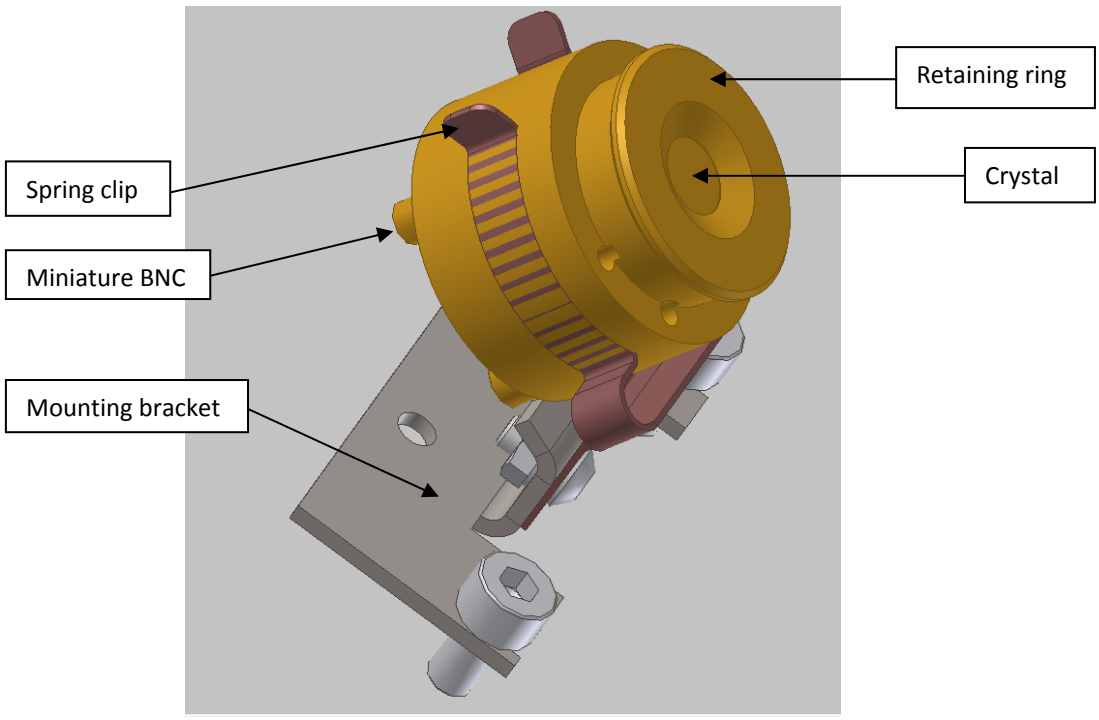


Figure 33 - FTM head components

3.7.3. Using the optional carbon evaporator

If the system detects the optional carbon evaporation unit is fitted an extra button appears on the main screen in the process area.

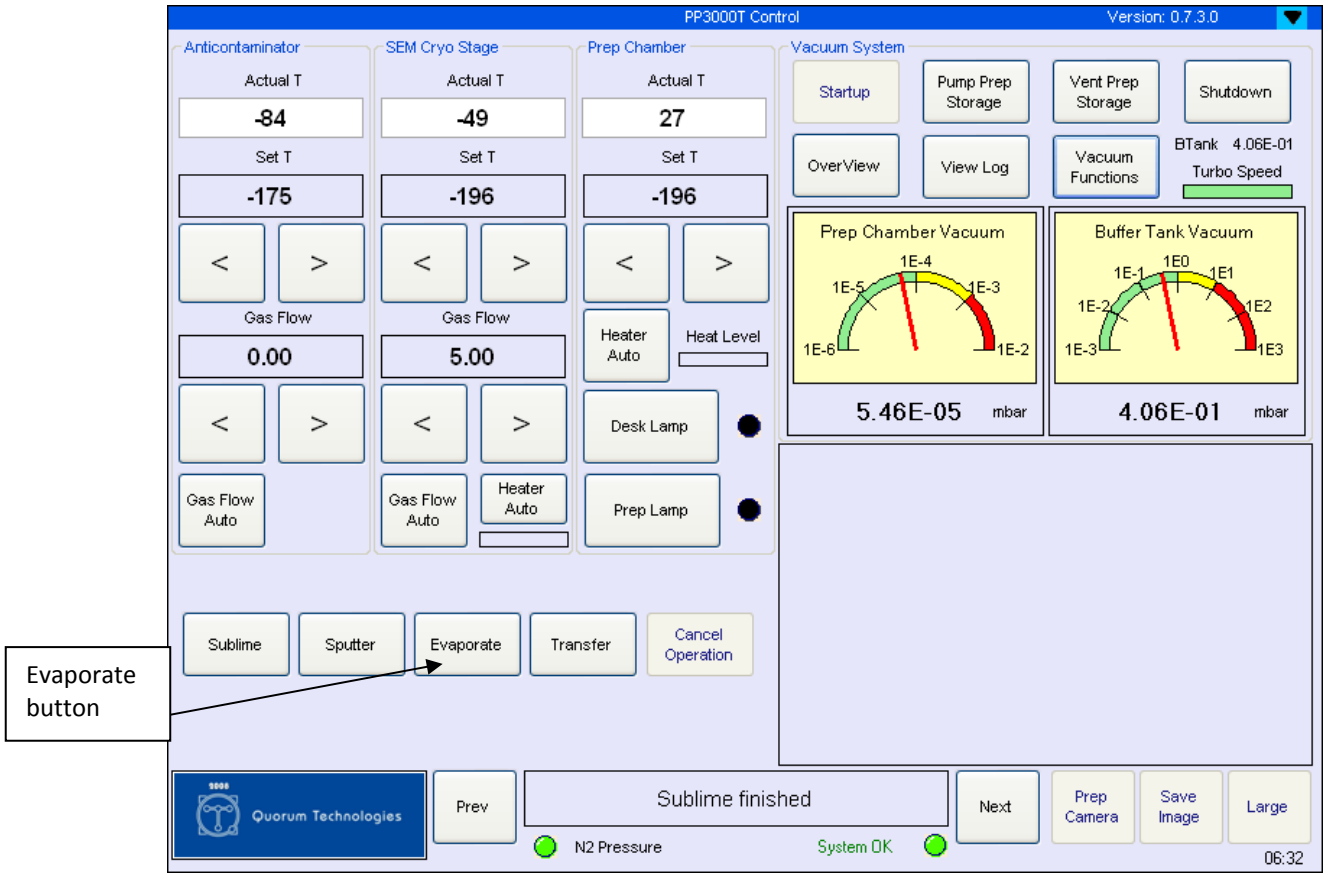


Figure 34 - Main screen with carbon option fitted

Touching the “Evaporate” button opens the evaporate sub menu

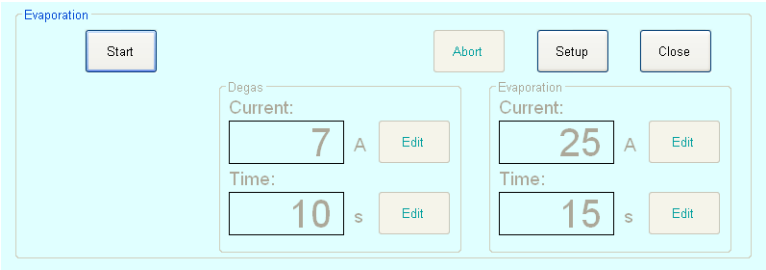


Figure 35 - Evaporate sub menu

The evaporation is fully automatic. Firstly, the carbon string is outgassed, during which time the specimen should be shielded using the micrometer knife. A message on the screen reminds the user to do this. Once outgassed the user us prompted to retract the micrometer knife. Once this is confirmed, the string will be rapidly heated until it fuses.

Should the user wish to alter the default parameters the “SETUP” button should be touched. This will activate the settings and they may be changed using the “EDIT” buttons.

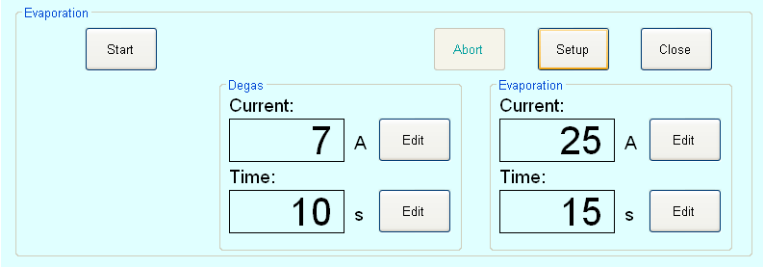


Figure 36 - Editing the evaporate parameters

3.7.4. Loading the carbon string

The carbon string is held in place by two spring loaded electrodes. Wrap the string around one of the electrodes under the spring loaded part. Make sure no frayed ends are sticking out. Gently pull the string across to the other electrode and wrap the string around it making sure it is under the spring loaded end. Gently ease the string back so it is not taut as this may lead to premature failure. Cut off any the excess length of string.

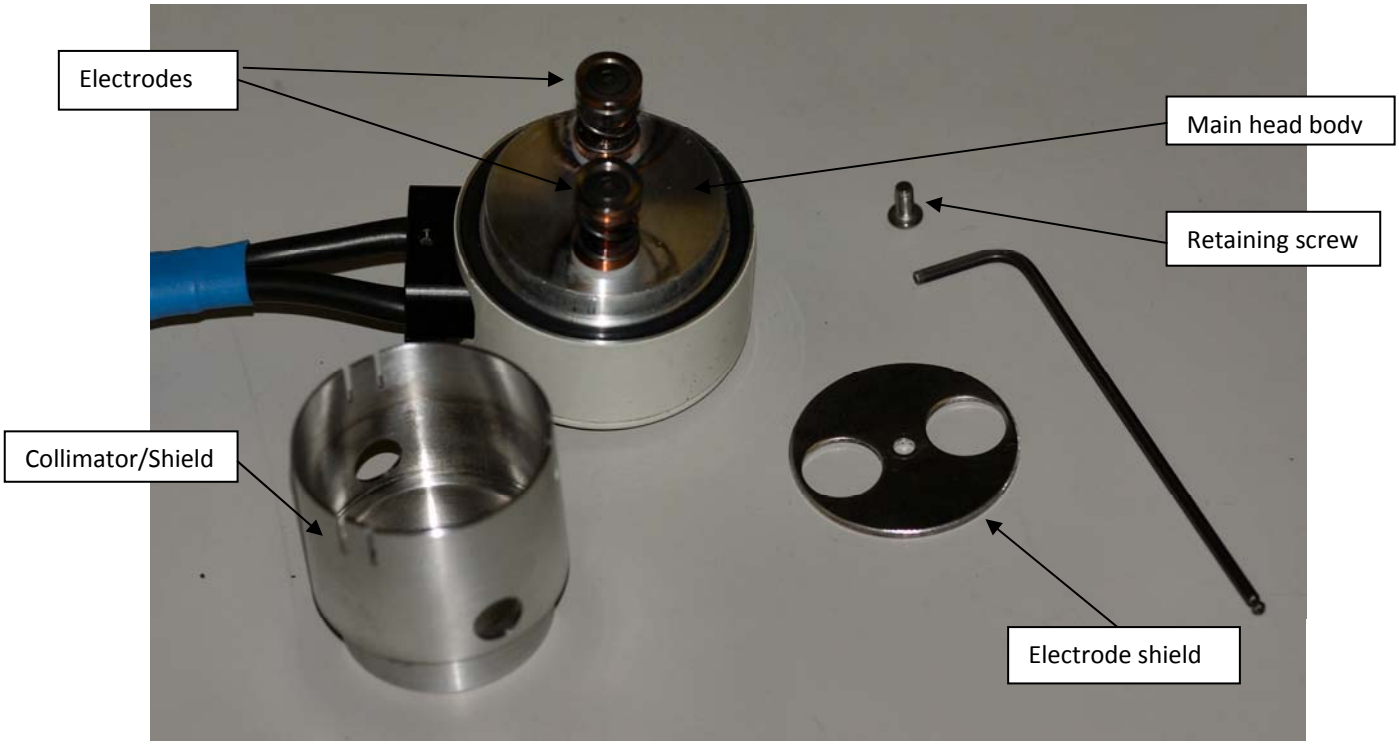


Figure 37 - Carbon head components

The carbon head will need periodic cleaning. To do this, vent the prep chamber and remove the head. The collimator/shield may then be pulled off to give access to the electrodes and electrode shield. Remove any remaining carbon string. Using a suitable Allen driver, unscrew the electrode shield retaining screw. Carefully clean all components with a lint free cloth and iso-propyl alcohol. Re-assemble making sure the electrode shield is centred on both electrodes.

Figure 38 - Assembling the carbon head

3.8. Imaging control

Where space allows, the system may be fitted with two webcams. One camera looks at the specimen whilst it is in the preparation chamber and the second is fitted inside the SEM chamber to provide a view of the specimen as it is loaded onto the SEM cold stage.

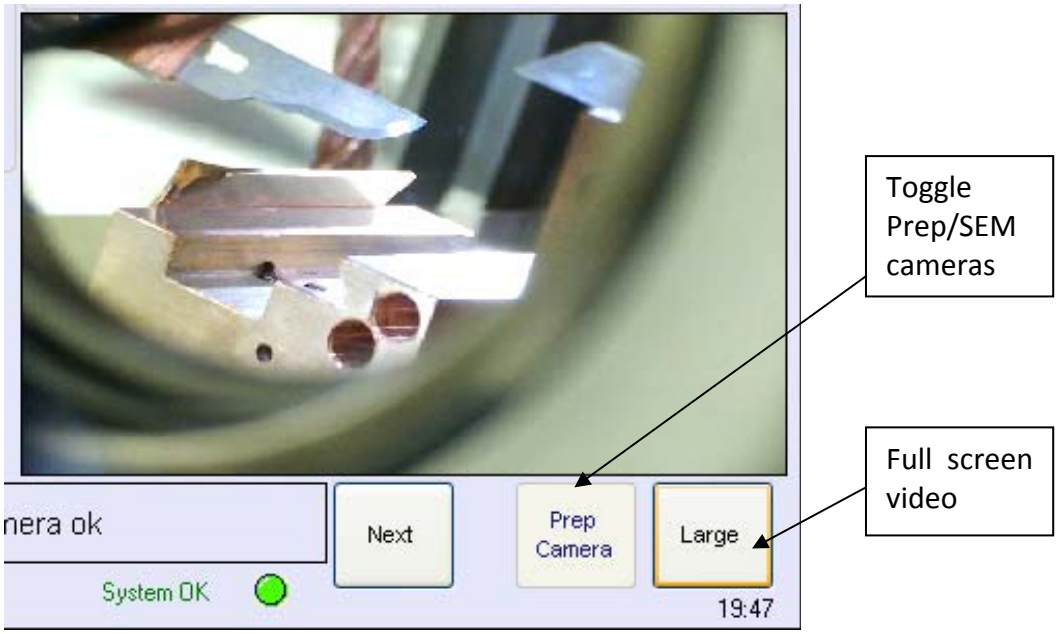


Figure 39 - Camera window

A toggle button switches between the two cameras and the "Large/Small" button switches between full-screen view and small window view.

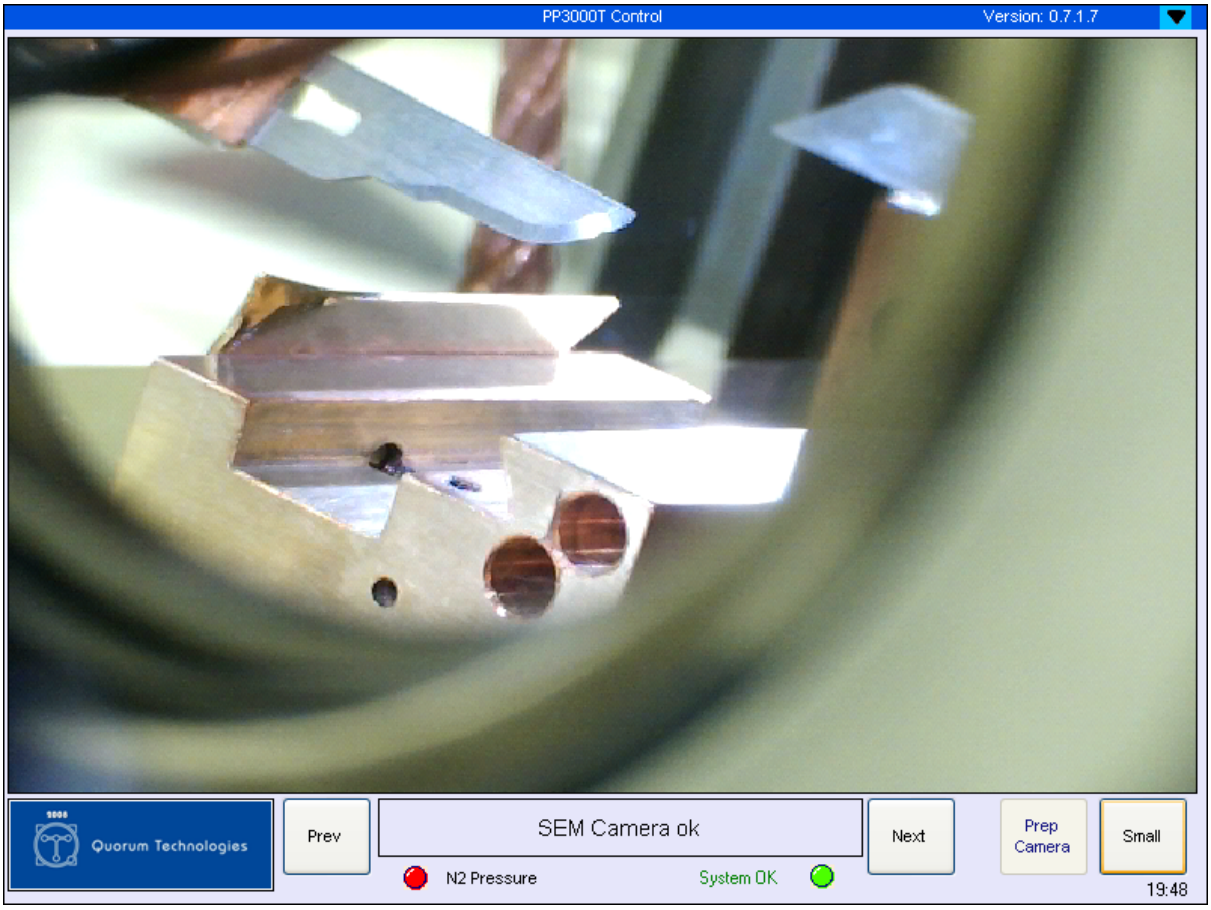


Figure 40 - Full screen video window

3.9. Messaging areas

There are several areas of the screen used to display messages to the user.

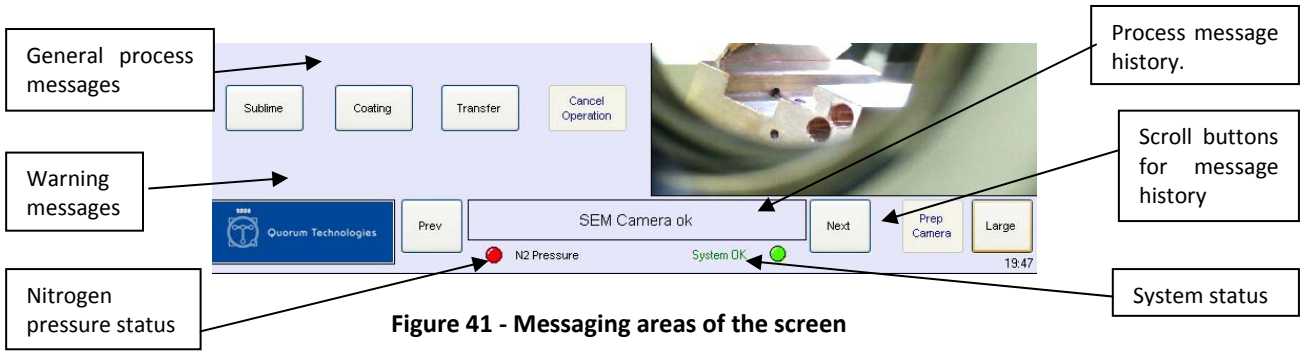


Figure 41 - Messaging areas of the screen

General process messages are displayed above the process buttons. Warning and error messages are displayed in the area below the buttons. A history of all the messages displayed since system start up is shown at the bottom of the screen. Touching the "Prev" and "Next" buttons allows the user to scroll through these messages. By touching the message itself the frame is expanded to show a scrollable window of all the messages.

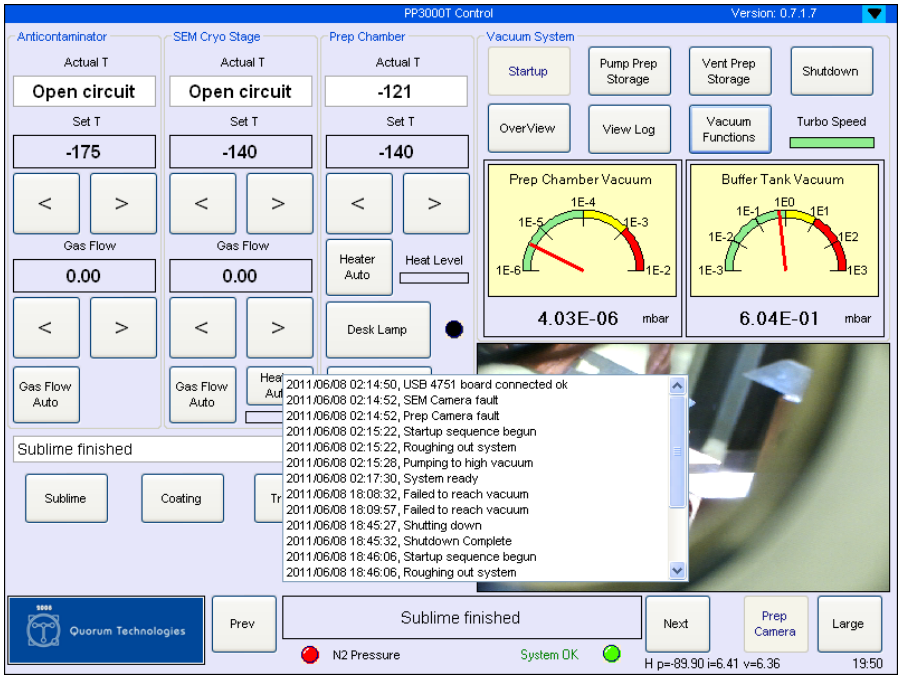


Figure 42 - Message history

3.10. Shutting down the software and access to reserved functions

Touching the logo in the bottom left hand corner opens a window where the software can be shut down and also gives access to the password protected area for diagnostics and service.

4. Running the system

4.1. Initial preparation

Before using the system it is advisable to pump out the vacuum isolated transfer line, especially after any long period without use. This is best done overnight, before the planned cryo session, but can be done during start up if necessary. See section 6.2 for details on how to pump out the line.

Make sure that:

a. microscope is under vacuum and fully operational. Set a low operating voltage, beam current and magnification. Move the SEM stage to the stored “cryo load” position.

b. nitrogen vent gas is turned on

c. liquid nitrogen dewar for the CHE3000 is filled but the heat exchanger not inserted

d. liquid nitrogen for slushing is available

e. nitrogen gas for cooling circuits is turned on

f. argon supply is turned on

g. specimen preparation tools etc are to hand

h. PP3000T preparation chamber is isolated from the microscope with both gate valves closed and locked

4.2. Start up

Switch on the PrepDek® using the on/off switch mounted under the rear left hand corner followed by the panel PC using the switch in the centre of its bottom cover.

Start the software by double tapping on the PP3000T shortcut on the desktop.

On power up and before the program starts, the LEDs should all briefly light up orange and the LED lamps come on as the self test runs.

Wait for the main control screen to be displayed. If all the processors and services are running the system status will show green.

Press the “START UP” button and select the “Auto Mode” or “Manual mode” option (Figure 17).
A window will pop up reminding the user to set the microscope to cryo operation.

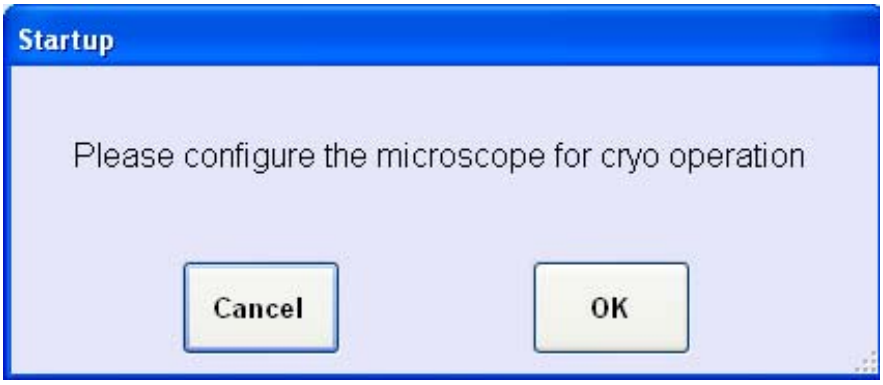


Figure 43 - Microscope configuration reminder

Once the user confirms by pressing “OK” another window pops up reminding the user to fit the pump out valve to the CHE3000.

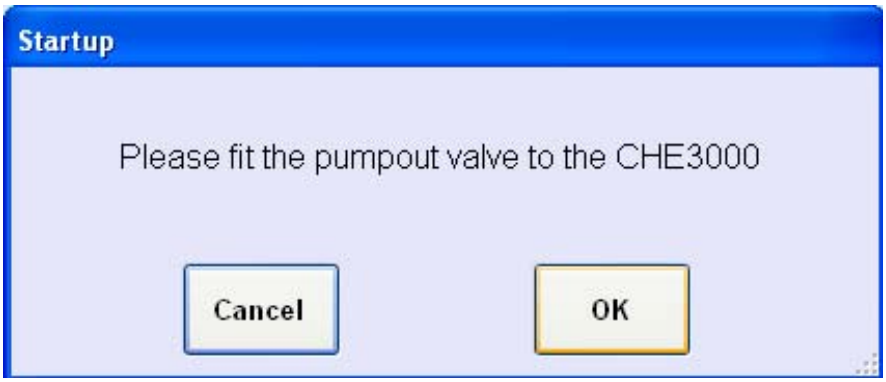


Figure 44 - CHE insertion reminder

On seeing “OK” the system will pump down the valve pumping line and check for leaks. If this test is passed another window will pop up requesting the user to open the pump out valve.

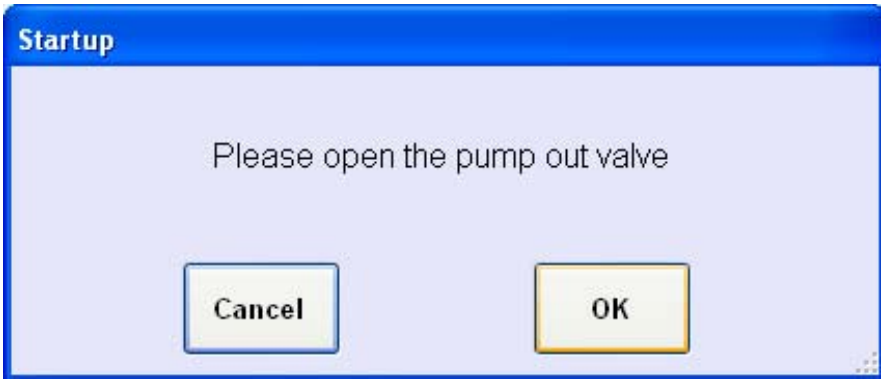


Figure 45 - Open valve reminder

The automatic pump down sequence will start. The rotary pump is switched on first and the vacuum in the lines checked for leaks. If this test is passed the buffer tank valve will open and the system will be roughed out. Again, if no leaks are suspected and the vacuum comes down to the set trip point, the turbo pump is started and the system pumped to high vacuum. This may take some time if the system has been at atmospheric pressure for any length of time.

4.3. Cool down

If the system has been started in “Auto” mode a message to cool down the preparation chamber will appear once the system has reached high vacuum. When the system sees the temperature of the prep chamber dropping, it will start a low (1L/min) gas flow through the CHE3000 to purge the lines. This is to make sure the lines are clean and free from any moisture which could cause a blockage in the gas lines. The purging lasts for 20 minutes. During this time the vacuum isolated line continues to be pumped. When purging is complete the user is prompted to close the pump out valve and remove it.

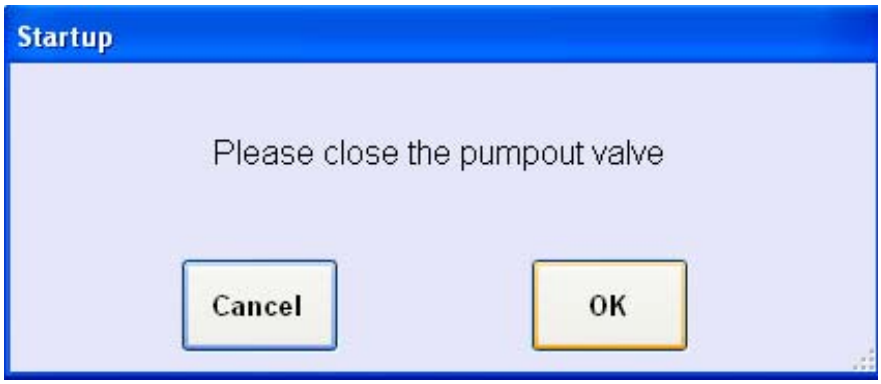


Figure 46 - Close pump out valve prompt

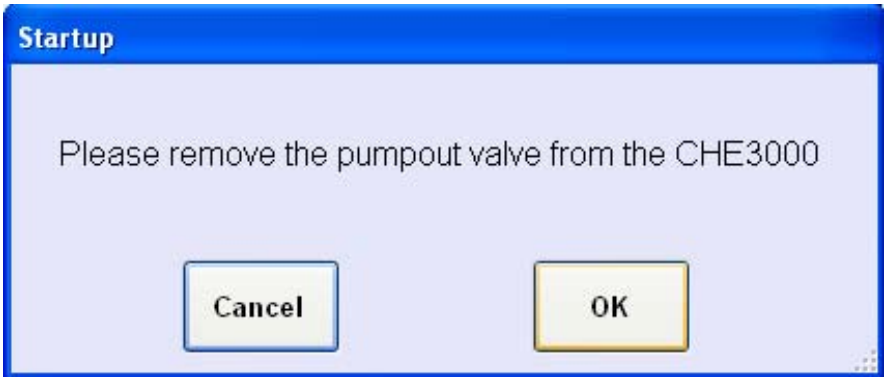


Figure 47 - Remove pump out valve prompt

Once this is confirmed the next prompt asks the user to insert the CHE3000 into the 12L dewar and increase the gas flow to 5L/min to rapidly cool the SEM cold stage and anti-contaminator. Once at the “Set” temperatures the gas flow can be reduced and, if required, “AUTO” pressed to start a PID control loop to maintain the set temperature. Often it is better to use manual control with a set flow to minimise gas usage.

The system will prompt the user that it is ready.

If non-default temperatures are required they can now be set.

4.4. Preparing a “standard” specimen

Make sure tools, specimen mounting, shuttles, stubs, hair dryer etc are available as needed

4.4.1. Making nitrogen slush

- Decide which slusher to use. Normally, for direct freezing of fresh material the smaller one.
- Fill the polystyrene beaker with liquid nitrogen taking care not to overfill
- Place the lid on the slusher pot and press the corresponding “SLUSH” button. Pressing the “SLUSH” button again will stop the pump.
- Wait for nitrogen slush to form (Nitrogen ice “pops” up)

Prepare the specimen and mount it on the specimen shuttle using whichever mounting technique is suitable. Fit the shuttle to the transfer rod bayonet.

4.4.2. Mounting methods

There are many ways to mount a specimen depending on the geometry and consistency. Below are a few examples.

4.4.2.1. Surface mounting

For leaf samples etc.

Roughen the stub surface with fine abrasive paper, place a small amount of mounting media (mixture of 50% Tissue-Tek®/50% colloidal graphite) onto the stub and lay the specimen on top of the mounting media.

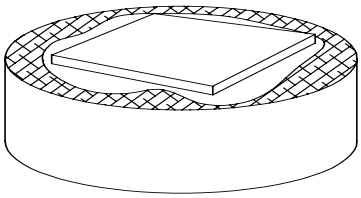


Figure 48 – Leaf sample preparation

4.4.2.2. Edge mounting

For edge observations and fracture

Roughen the surfaces of a stub with fine abrasive paper. Mount the specimen on edge secured with mounting media or mount the specimen standing on edge in a machined slot, secured with mounting media. This is important if the specimen is to be fractured.

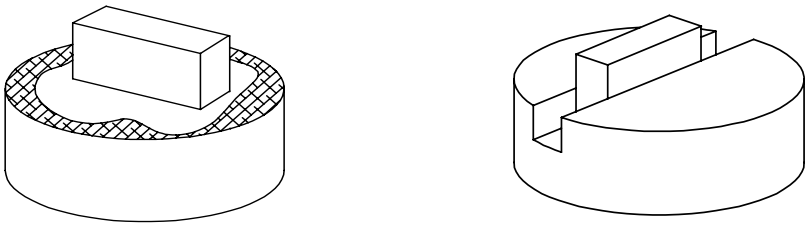


Figure 49 - Edge mount preparation

4.4.2.3. Filter mounting

For liquid suspensions

Secure the filter paper to the stub with four droplets of mounting media. Secure the membrane filter to filter paper base using mounting media. Pipette liquid suspension onto membrane filter.

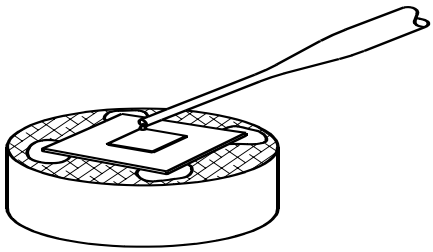


Figure 50 – Filter mounting preparation

4.4.2.4. Hole mounting

For thicker emulsions and liquids (i.e. oil, toothpaste)

Use a hole or holes drilled in stub to locate thicker emulsions.

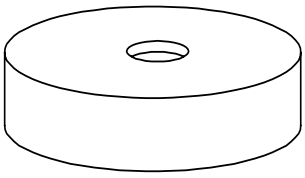


Figure 51 – Hole mount preparation

4.4.2.5. Film emulsion mounting

This is useful when the specimen is small and would be obscured by Tissue-Tek or when specimens need to be recovered. Specimens need to be damp to use this method (i.e. Nematodes).
Mount exposed unused film with the emulsion side up to the stub with mounting media. Lay the damp specimen on the surface. The specimen dampness slightly dissolves the film emulsion allowing the specimen to adhere to the film surface.

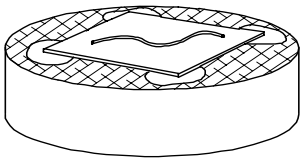


Figure 52 - Film mount preparation

4.4.2.6. Liquid film mounting

Liquid specimens can be thinly spread on a plain stub which has been roughened with fine abrasive paper.

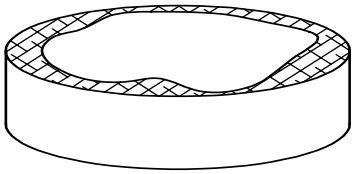


Figure 53 - Liquid preparation

4.4.2.7. Rivet mounting

Use rivets for liquids and when specimens need to be frozen off the stub to achieve fast freezing rates. The rivet is placed in a hole and filled with liquid prior to freezing, or if the specimen needs to be frozen rapidly off the stub, 2 rivets are held together, filled with liquid and plunge frozen prior to mounting in the rivet holder. Rivets (part # 39996300) will push fit into the two holes of the E7449 multi-stub supplied with the system.

Figure 54 - E7449 stub

Figure 55 - Rivet mounting preparation

Other specimen stubs and shuttles are available (see section 8 PP3000T shuttles and stubs). Users can also modify blank stubs (E7402 – supplied with the system) with slots or holes.

4.4.3. Freezing

Once the specimen is mounted, press and hold the "VENT" button to admit dry nitrogen gas to the nitrogen slushing pot. Remove the lid and plunge the specimen shuttle under the nitrogen surface. Wait for the nitrogen to stop boiling. Slide the transfer device down the insertion rod, keeping the specimen shuttle below the surface of the liquid nitrogen until the transfer device engages with the top of the slusher and the airlock pin enters the hole in the slusher lid. Press the "SLUSH" button again. Just before the nitrogen turns to slush again, withdraw the insertion rod into the transfer device and close the small swivel valve by turning the black knob on the back of the transfer device a few turns. Once the valve is closed press and hold "VENT" until the transfer chamber can be removed. Replace the Perspex cover on the slushing pot.

4.4.4. Transferring to the preparation chamber

Mount the transfer device on the preparation chamber airlock making sure the pin locates in the guide and the device is square to the flange.

To evacuate the airlock:

- Press the airlock "PUMP" button.
- The "PUMP" LED turns red, the airlock valve opens and the rotary pump starts pumping down the airlock.
- Open the small swivel valve by unscrewing the black knob about 3 turns.
- Wait for green LEDs on "PUMP" and airlock valve. This means that the airlock vacuum has reached the set point and the interlock solenoid has been released.

- Rotate the airlock valve control knob anticlockwise 90° and pull it downwards to open the valve.
- If the valve is not opened within 30 seconds the solenoid re-locks and the pump down sequence must be re-started.
- After the valve has been opened the solenoid re-locks after 5 seconds
- Slide the insertion rod into the preparation chamber and slide the specimen onto cold stage.

4.4.5. Fracturing

Two means of producing a fracture are provided; a front mounted scalpel-bladed probe and a top-mounted micrometer-advanced knife. Both are actively cooled by copper braids connected to the cold block in the preparation chamber. The scalpel blades can be exchanged and any size #5 blade may be used depending on the application or user preference. This blade can be used to fracture thinner specimens, to flick pieces of specimen away, to open complementary fracture holders and to knock off rivets.

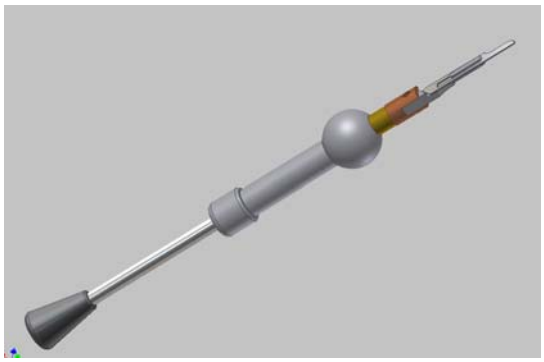


Figure 56 - Front mounted probe

The micrometer advanced knife is used for thicker specimens and the height of the blade is adjusted by turning the knurled knob below the actuating lever. The knife should be raised before swinging back otherwise it may smear the top of the specimen.

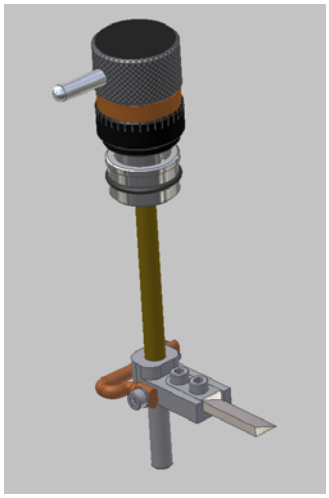


Figure 57 - Micrometer advanced knife

4.4.6. Sublimation

Once a fresh surface has been exposed it is usually necessary to sublime the surface. The sublime button is touched and brings up the sublimation screen. Once the desired profile has been selected either from one of the stored recipes or a new recipe defined, the user needs only to press “Start” for the process to run automatically (see the software section 3.6).

4.4.7. Coating

Following sublimation, the specimen will usually need to be coated with a thin conductive film of metal. This is done via the “Coating” button. Pressing this button calls up the coating screen and again, once the required current and time have been selected, pressing “Start” runs the process automatically (see the software section 3.7).

4.4.8. Transferring the specimen into microscope

Following coating, the specimen is transferred into the SEM chamber and is mounted on the SEM cold stage. To initiate a transfer the “Transfer” button is touched. If the preparation chamber pressure is low enough the SEM gate valve solenoid will be released and the gate valve LED will turn green. The gate valve may now be opened by rotating the black knob clockwise and pulling down to the end stop. As the SEM gate valve opens the SEM chamber lamp will automatically be turned on.

The specimen is now withdrawn from the preparation chamber cold stage by gently pulling back on the transfer rod and then advanced through the preparation chamber and SEM gate valve. Looking through the transfer device window or at the CCD image, slide the specimen dovetail onto the SEM cold stage. Rotating the insertion rod 90 degrees will release the bayonet and the insertion rod may be retracted into the transfer device. The SEM gate valve should now be closed and the solenoid will lock and the SEM lamp will extinguish. Close the transfer gate valve.

The specimen should be imaged and if the preparation is successful the transfer device should be removed from the preparation chamber to reduce any induced vibration. This is done by pressing and holding the “Vent” button on top of the airlock and once vented, lift the transfer device off the airlock and place in the storage tube.

4.4.9. Removing the specimen

Drive the SEM stage to the cryo load position. If the transfer rod has been removed from the preparation chamber it must be re-fitted and evacuated as per inserting a specimen. Open the gate valve and check the prep chamber pressure recovers. Press “Transfer” to release the SEM column valve and open the valve. Slide the insertion rod fully into the

SEM and engage the bayonet into the specimen shuttle. Slide the rod fully back into the transfer device; close the SEM valve followed by the prep valve. Partially close the small transfer device cover to make sure it clears the prep valve body. Vent the airlock and remove the transfer device. Remove the specimen shuttle, warm and dry the end of the insertion rod.

4.5. Advanced specimen handling pot

For clarity the images below are shown without the main cover and with no liquid nitrogen.

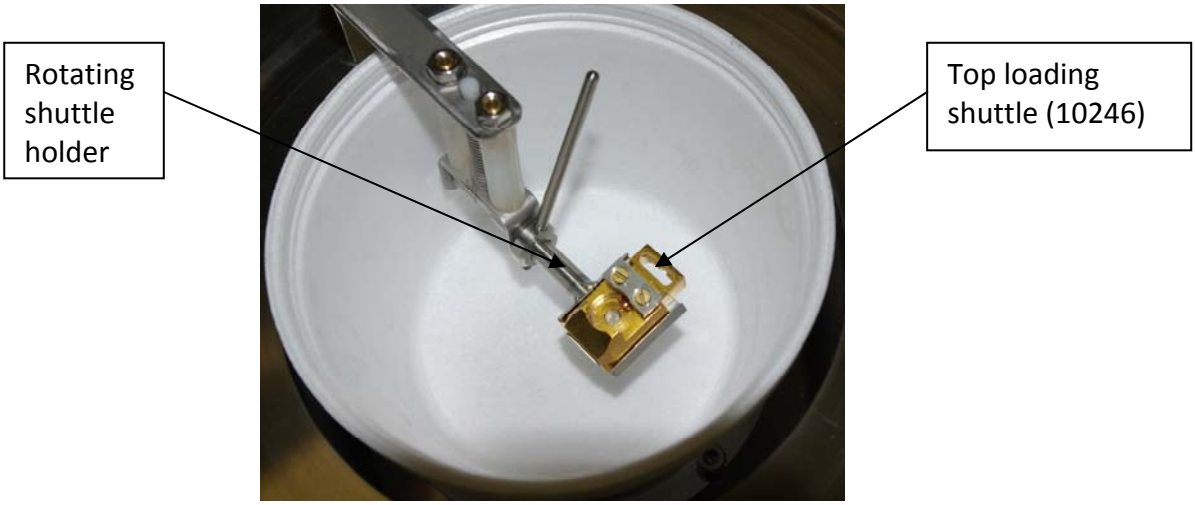


Figure 58 - Loading position

Firstly, load a top loading shuttle* into the rotating shuttle holder and turn it so that it is horizontal (Figure 58). Fill the polystyrene cup with liquid nitrogen, taking care not to overfill. Place the Perspex cover onto the pot and press “Slush”. Once nitrogen slush forms the pot may be vented and the cover removed.

* Top loading shuttle (10246) is supplied with the system. Other types can be purchased.

Figure 59 - Pick up position

Transfer the pre-prepared specimen into the pot and load it into the shuttle. Rotate the shuttle holder so that it is vertical (Figure 59). Pre-cool the tip of the transfer rod in the liquid nitrogen and locate the bayonet fitting into the shuttle (Figure 61). Rotate 90 degrees to lock onto the shuttle. Slide the transfer device down the insertion rod and locate it on top of the pot cover (Figure 60). Press “Slush”. Just before slush re-forms, slide the insertion rod back into the transfer device and close the small swing valve. Continue the transfer as normal.

Figure 60 - Transfer device fitted

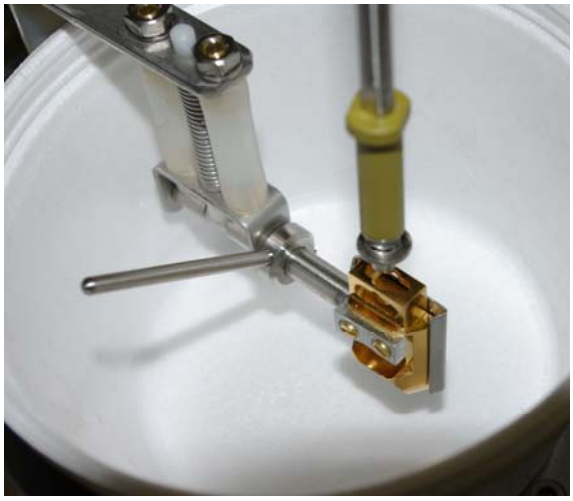


Figure 61 - Picking up shuttle

Figure 62 - View of pick up

5. Shutting the system down

Drive the SEM stage to the cryo load position

Remove the specimen from the microscope

Remove CHE3000 from the 12L dewar

Touch “Shutdown” on main screen

Wait for “Shutdown complete” message

Turn off PC and PrepDek®

Turn off the argon and nitrogen gas supplies

6. General Maintenance

6.1. Pumping the transfer device storage tube

A position is provided in the PrepDek® where the transfer device can be stored under vacuum to keep it clean and free from moisture.

Figure 63 - Transfer device fitted on storage tube

Place the transfer device in the storage tube and slide the rod down to the stop. Touch the “Pump Storage Tube” button on the screen in the “Vacuum” area. The storage tube will be pumped down to a set vacuum and then the valve closed to leave it under vacuum. To vent the device, just touch the “Vent Storage Tube” button.

6.2. Pumping the vacuum isolated pumping line

In order to maintain a good vacuum in the vacuum isolated cold gas delivery line, it needs to be evacuated periodically. Pumping also regenerates the sorb material in the body of the line.

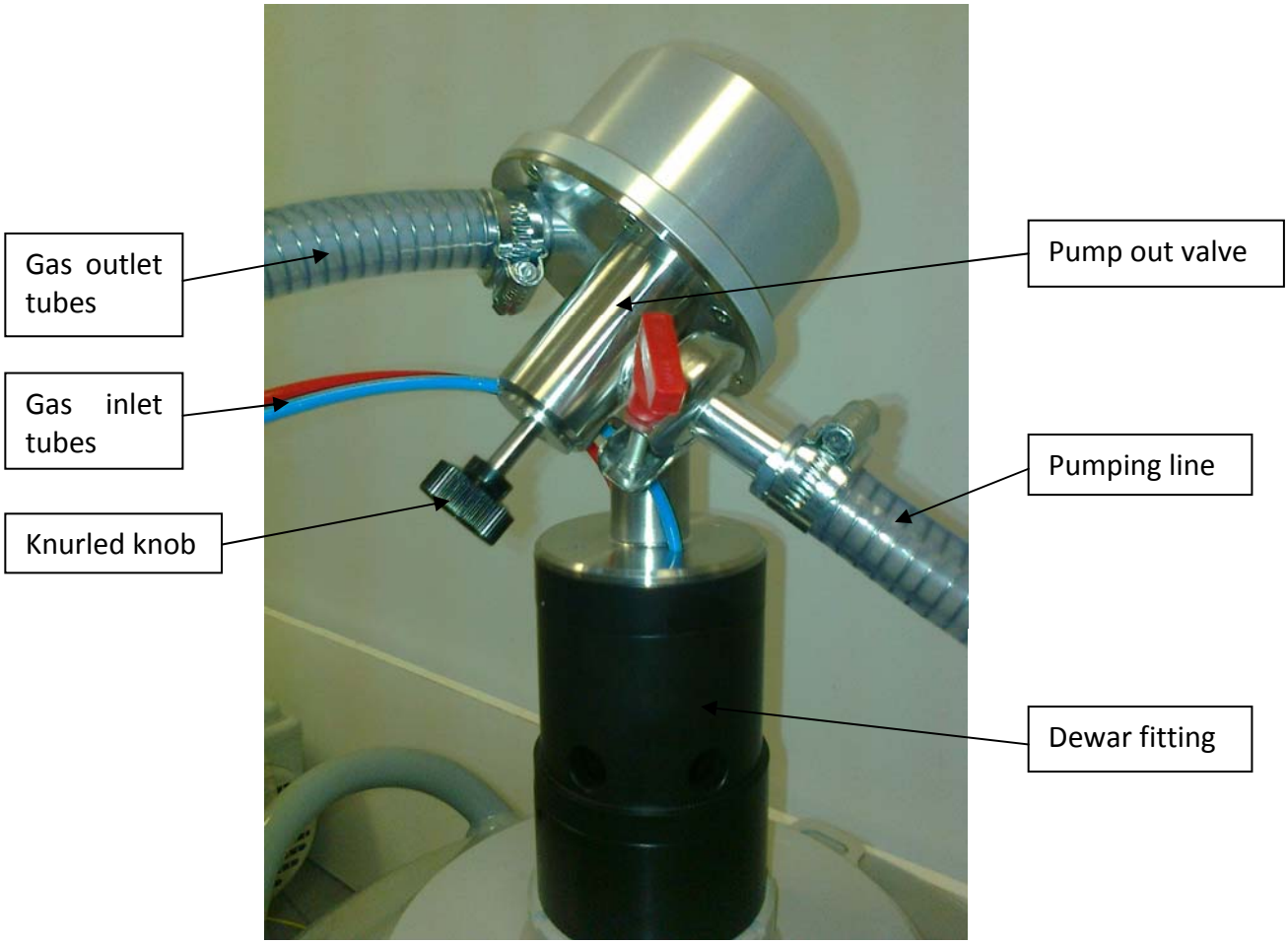


Figure 64 - Pumping the vacuum isolated line

A flexible pumping line with a fitting which slides onto the pump-out port of the vacuum isolated line is supplied. The fitting has a threaded shaft which engages in a piston sealed plug in the port. Slide the fitting over the pump out port and screw the shaft into the plug for about five turns. As the vacuum isolated line will only be pumped out occasionally, the control buttons to pump it are found under the "Vacuum functions" button. Touching this button opens a window with the controls. Firstly, check there are no leaks in the line or connection by touching the "Pump vacuum isolated line" button. The right hand vacuum gauge will then indicate the pressure in the line. Make sure this comes below $1e^{-2}$ mbar. If this is the case then gently pull the threaded shaft out of the pump out valve until it comes to a stop. The line should then be left to pump for several hours or overnight. A vacuum of $5e^{-2}$ mbar should be reached.

Once the line has been pumped, press the threaded rod back into the pump out tool and unscrew it until it disengages from the piston sealed plug. The pumping line may now be vented and removed from the pump out fitting by touching the "Vent vacuum isolated line" button.

When not pumping the vacuum isolated line the line should be removed from the CHE3000 to avoid any vibration being transmitted to the microscope.

6.3. Lubricating the vacuum transfer device rod

The stroke of the transfer device rod is quite long and it requires periodic lubrication. This should be done at the beginning of each cryo session and as necessary during a long session if the rod does not slide easily and freely. A small container of high vacuum compatible lubricant is supplied with the installation kit (see Figure 65).

Figure 65 - Lubricant for transfer rod

Pull the insertion rod fully out and using a soft, lint-free tissue, carefully wipe the rod to remove any old lubricant and dirt which may have accumulated.

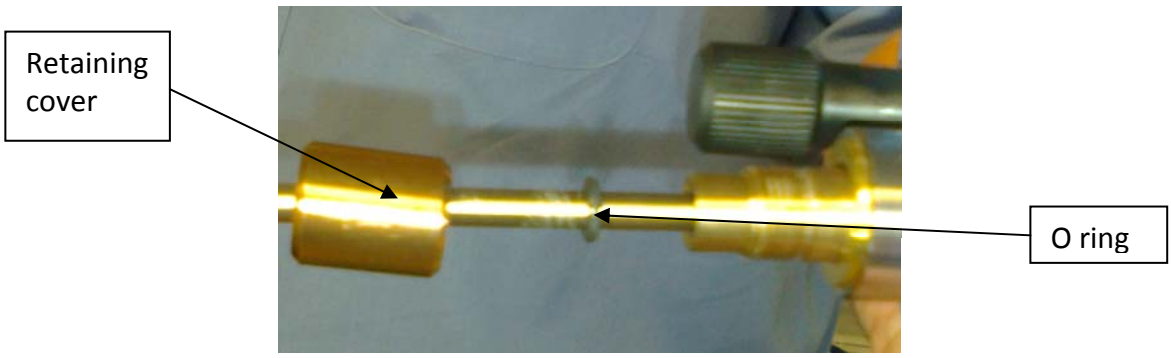


Figure 66 - Rear of transfer device

Unscrew the retaining cover (see Figure 66) from the back of the rod ball joint. Pull the rod sharply outwards to reveal the 'O' rings.

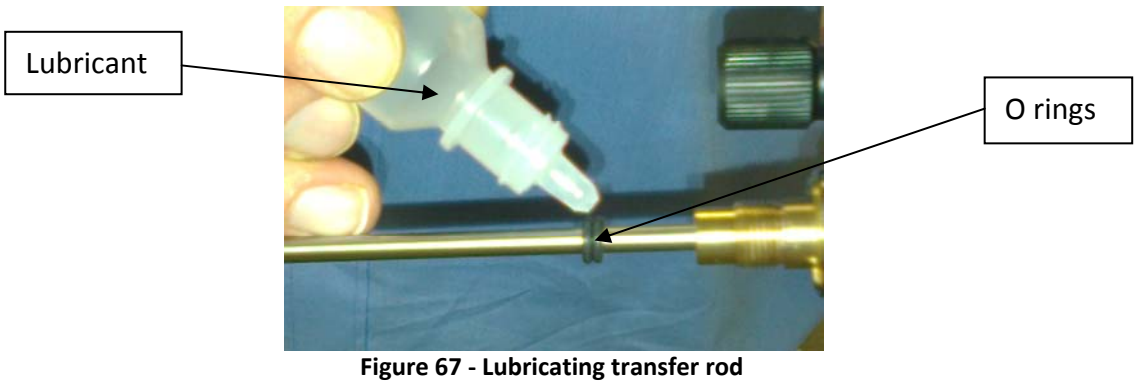


Figure 67 - Lubricating transfer rod

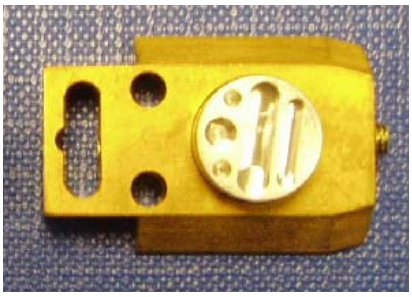
Place a drop of lubricant on the rod near the ball joint at the back of the transfer device. (see Figure 67) Replace the retaining cover. Slide the rod backwards and forwards a few times to distribute the lubricant along the rod.

7. Troubleshooting

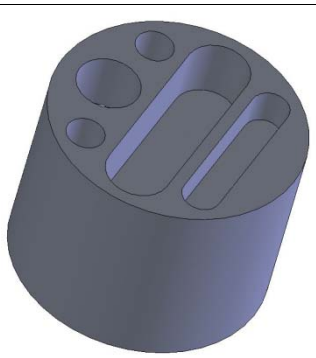
The system is designed to be mostly self diagnosing should there be a problem. Any malfunction of a main system component such as temperature control unit, power supply etc will be displayed in the message window at the bottom centre of the screen. Any loss of communications will also be displayed here. The system also logs data to three files in the Quorumtech folder in Program Files. These can be useful for diagnostic when contacting the Quorum Service department.

8. PP3000T shuttles and stubs

There are many styles of shuttles and stubs available for the PP3000T. Some of the most common are shown below:

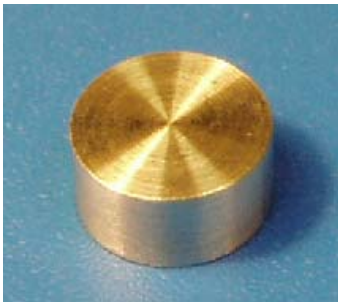


AL200077B
Specimen shuttle (with E7449 universal stub)



E7449
Universal cryo stubs (multi-stub)

E7449
5 universal cryo stub – packet of five



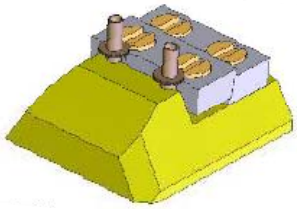
Blank specimen stub (5mm high)
E7402 = aluminium
E7403 = copper

E7407
Copper stub with 1mm slot

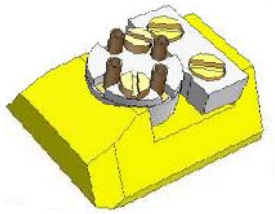
E7406
Copper stub with 3mm slot

E7404
Clamping stub

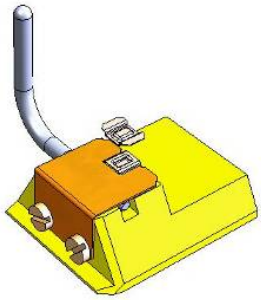
328116510
Freezing rivets



10247
Rivet shuttle (bayonet fitting)



10246
Shuttle for 10mm stub shown with E7433 rivet stub



10245
Shuttle for freeze fracture planchettes

9. Maintenance

With the exception of the 'regular maintenance tasks', the service and repair of the PP3000 should be carried out by qualified service engineers.

Warning, lethal voltages are present in this instrument; disconnect power to the instrument when carrying out service tasks as indicated in the relevant section of this manual.

Recommended tools:

Set of metric Allen keys, 1.5 to 8mm
Long reach 'magic ring' M3 Allen key
Dewar top nut spanner
5/16"-1/4" spanner 395020220
Mini vacuum cleaner
LED hand light
21mm open ended spanner
22mm open ended spanner
½" paint brush
Peg spanner or circlip pliers (transfer device)
Dental mirror
Screw retaining tool
Small size nozzle to suite vacuum cleaner (Vacuum cleaner not supplied)

9.1. Preparation chamber

Please note: the preparation chamber and many of the parts of the cryo system are manufactured from light aluminium alloy. This material is highly suitable for this application and with care the instrument will last many years. It is however easily damaged if mistreated. Great care must be taken of seal faces and in the tightening of screws.

Figure 68 - Using an Allen key

Without exception, screw threads into aluminium must not be over tightened, and it is acceptable to use only the short arm of a standard Allen key for tightening.
Mistreatment of the chamber components will void warranty cover.

9.2. Cleaning the preparation chamber

This is a regular maintenance task

9.2.1. Introduction

Cleaning of the preparation chamber is required in order to keep the instrument operational. The frequency of this cleaning will be determined by the amount of use and sample type.

9.2.2. Reasons for cleaning are:

To remove debris from sample fracture
To retrieve sample mounting rivets (if used)
To remove build-up of sputtered material on internal chamber surfaces, viewing windows and chamber lights

Cleaning of the preparation chamber will normally be done at the end of a cryo work run, in readiness for the next cryo work run, and having allowed the instrument to come up to ambient temperature.
It will be assumed that the instrument is being pumped and is in good working order.
Clean gloves must be worn throughout the maintenance routine to avoid contamination of vacuum surfaces.
The cleaning process is simple and should not give any problems if conducted in a methodical manner. Prepare by placing a soft mat on the microscope work top below the preparation chamber and have space enough to lay out the parts. It is a good idea to have small dishes to keep screws safe.
Some of the work may need to be done away from the cryo chamber and microscope, so prepare workspace in advance.

Two levels of chamber cleaning are possible. Cleaning through the front window port will allow cleaning of accessible surfaces, removal of debris, used rivets and dropped samples. This will be referred to as the ‘Short method’, and may be required on a weekly basis depending on instrument usage.

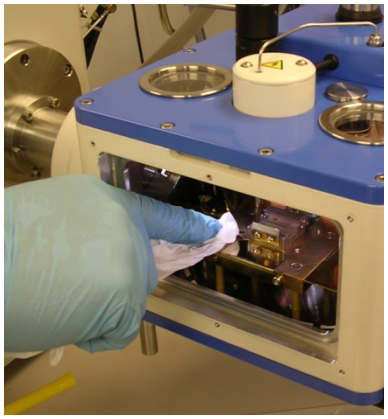


Figure 69 – Working though the front window

A more thorough cleaning will be possible if the cold stage and liquid nitrogen dewar is removed from the chamber body. This will be referred to as the 'Thorough method', and may only need to be carried out annually depending on instrument usage.

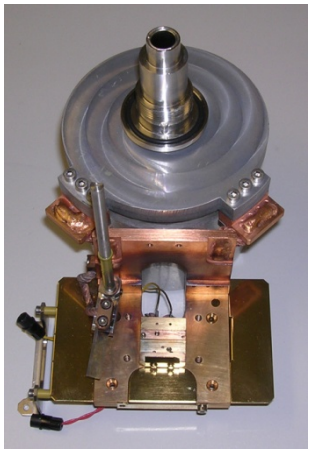


Figure 70 - Cold stage and dewar

Depending on the installation, this should be possible without removing the chamber from the microscope, but will involve removal of either the top or bottom plate of the chamber, again depending on the installation configuration. See details in the relevant section.

9.3. Chamber cleaning -short method

This is a regular maintenance task that should be performed by the instrument user.

9.3.1. Venting the preparation chamber

During the chamber venting you should monitor the microscope vacuum level. If the microscope pressure is increasing, this will indicate a leak across the SEM gate valve, which will need to be resolved. In the case of a pressure increase it is recommended that controlled venting of the microscope chamber should be performed.

The nitrogen gas supply must be connected and set to deliver the pressure required to enable the ‘N2 pressure’ indicator of the PP3000 main operating page.
Refer to the instrument operation instructions to vent the system and select the ‘VENT SYSTEM’ option.

The turbo pump will be switched off, and after a short time the vent valve will open to vent the chamber. The indication that the chamber has vented, is that the sputter head will now be free.

Cancel the venting operation if it has not cancelled automatically.

Remove the front window retaining bezel. This is held by six M3 countersunk screws. Hold the glass and bezel as you remove the last screw and do not let the bezel fall against the probe feed through.

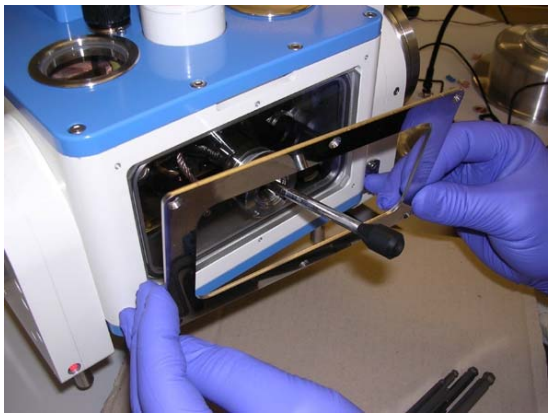


Figure 71 - Removing the front bezel

Holding the glass window in place, put the bezel to one side.
Now draw the window out ‘squarely’. The window is quite thick and if it turns at an angle as it comes out it may jam and this will cause the glass to flake and chip.
If this occurs, a replacement window will be required.

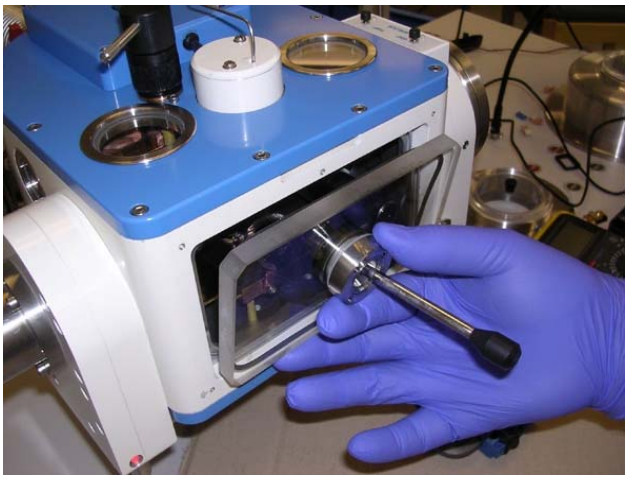


Figure 72 - Front window removed

You will need to have the 2.5mm hexagon wrench ready as the window is withdrawn, so that you can detach the cooling braid for the front window knife.

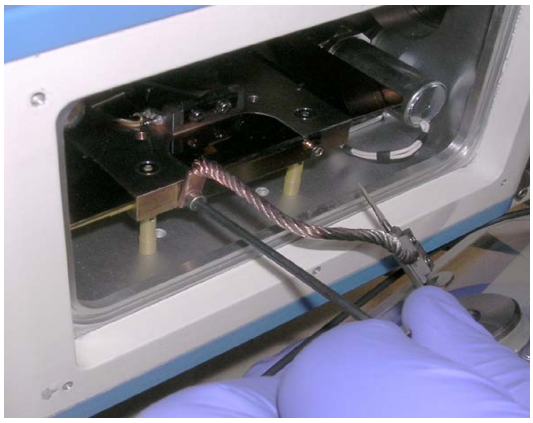


Figure 73 - Knife braid screw

Place the window plate with its knife manipulator on a soft surface. It should be noted that the knife manipulator, fitted to the front window has a scalpel blade as its cutting edge.

There is a high risk of personal injury!!!

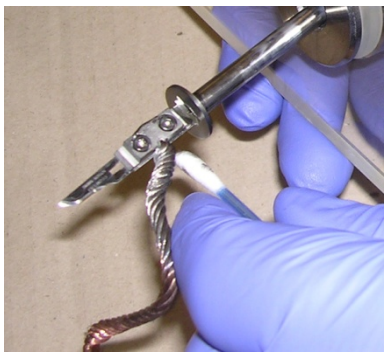


Figure 74 - Knife and braid

You now have access to the chamber though the front opening.
You may decide to remove the sputter head at this stage although it is not entirely necessary.

To do this, you will need to release the argon gas feed capillary. This will require the 5/16” spanner.

Figure 75 - Argon union

There is a cover over the top of the LN2 dewar, through which the sputter head cable passes.
The cover is retained by four M3 countersunk screws. You will need a 2.5mm hexagon wrench for this.

Figure 76 - Top plate cover removal

Lift the dewar top cover and sputter head away from the top of the preparation chamber, and place on a soft surface to avoid damage. Take care to retrieve the seal ring from the dewar neck above the cover plate.

Please note: the sputter head is of the 'Magnetron' type and as so, has a powerful magnet behind the target foil. Take care to keep magnetic particles away from this.

Use tweezers to remove dropped samples and discarded rivets from any accessible surfaces.
If possible, use a small vacuum cleaner pipe to remove debris from the cold shield top surface and from the lower front of the chamber.

Figure 77 - Chamber cleaning with vacuum adapter

Using cotton buds, remove the worst of the stray sputter coating from the cold shield, taking care not to leave any debris from the cleaning materials.

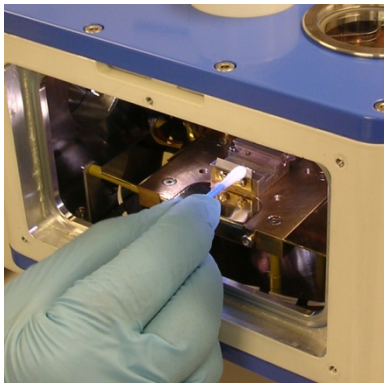


Figure 78 - Using cotton bud to clean the chamber

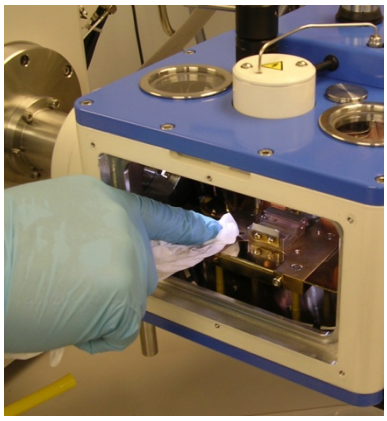


Figure 79 - Cleaning chamber with tissue

Clean the cold stage and cryo shields with lint free tissues and cotton buds.
Use clean alcohol.

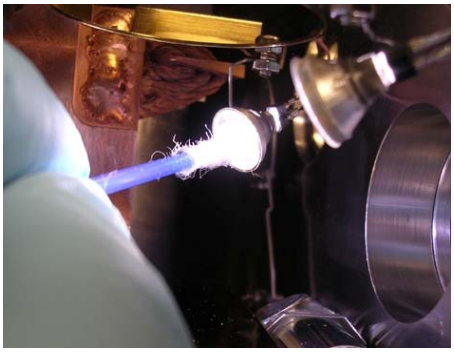


Figure 80 - Cleaning the LED cover glasses

Clean the cover glasses of the LED chamber lights with cotton buds and alcohol. Be very gentle with these lights as it is possible to dislodge the cover glasses.

Inspect the inside of the chamber to be sure that there are no damaged cooling braids or wires. Check the operation of the chamber lights, and knife mechanism.

At this stage you should re-fit the sputtering head, if it has been removed.
Inspect the o-ring and place the sputter head through the top plate, place the dewar top cover in place taking care to position the sputter power lead clear of the other parts under the cover. Connect the argon feed tube.
Fit the four screws that retain the dewar top cover.
Now, press down on the sputter head with your finger and view the sputter head from below with a dental mirror to ensure that there is clearance between the sputter head and the top cryo shield. Any contact will cause a cooling breech, and must be rectified.

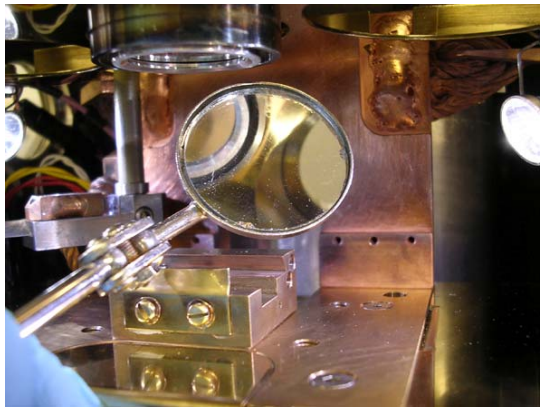


Figure 81 - Checking the sputter head clearance

The front window may now be re-fitted but it is likely to need cleaning first. Sputtered material will have coated the inside of the window which will greatly reduce visibility.
Take great care in handling the window as its edges will be very easily chipped. It should once again be noted that the knife manipulator, fitted to the front window has a scalpel blade as its cutting edge. **There is a high risk of personal injury!!!**

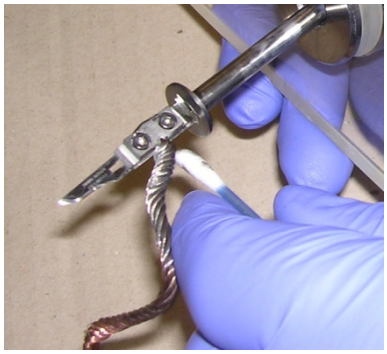


Figure 82 - Cleaning the knife

Make sure that the window seal 'O'ring is clean and correctly located in its groove.
Holding the window adjacent to the window opening, extend the knife manipulator into the chamber and re-connect the knife cooling braid.

Slide the window into place and hold in position.
Place the window bezel in place and fit a screw top and bottom so that the window is held in place.
Re-fit the remaining screws and gently tighten them in turn until they are all secure. Please note that that this does not mean 'too tight'.

Once the chamber is sealed check:

That the prep chamber lights are working
Check that there is a sensible temperature reading for the prep chamber stage (software test function required)
Check that the side window sputtering shutter works
Check that the stage heater is working
To do this, set a temperature for the stage that is above the read temperature and set the heater operation to AUTO. You will see the heat power reading bar show an indication of applied power and the temperature of the stage should rise.

If all of these functions are working satisfactorily, the chamber may be evacuated by selecting the 'START' option and choosing CRYO or A/LOCK mode.

Observe the vacuum recovery and turbo speed. The turbo should reach 100% and a pressure of XXx10-X within XX minutes.
Failure to reach this vacuum level or failure of the turbo pump to reach full speed will indicate a vacuum leak.

9.4. Chamber cleaning - thorough method, top access

This is a "regular maintenance task", that should be performer by the instrument user.
This procedure will allow full access to the internal parts of the preparation chamber but should only be required if the chamber has become excessively 'soiled' or in order to rectify faults that may have occurred.

9.4.1. Remove the top plate

Isolate the supply of argon gas to the PP3000.
Loosen the argon supply union at the back of the chamber below the main pumping pipe connection with the 5/16” spanner and disconnect the capillary tube.

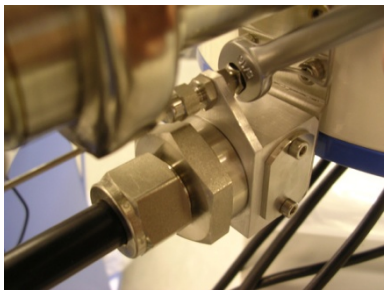


Figure 83 - Argon union loosening

Begin by performing the ‘short method’ cleaning procedure to the point where the sputter head has been removed but do not carry out any cleaning at this stage, cleaning will be done once the chamber has been further dismantled.

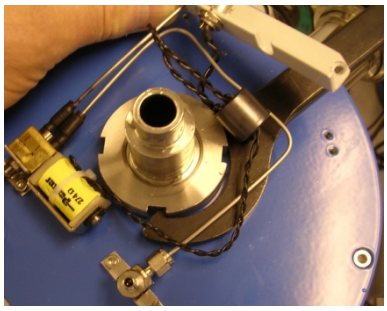


Figure 84 - Loosening the dewar nut

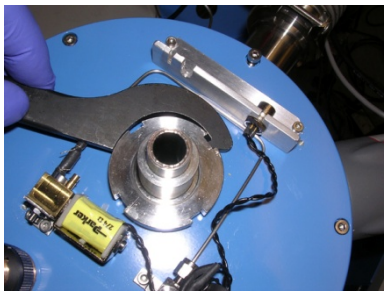


Figure 85 - Loosening dewar nut 2

Loosen the dewar to nut with the special spanner supplied with the instrument tool kit.
You may need to remove the bracket that holds the pipes and electrical plug in order to gain access to the dewar nut.
Remove the nut and the plastic washer that will be found under it.

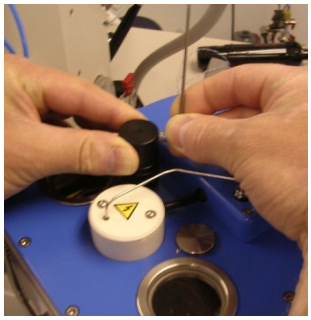


Figure 86 - Knife knob screw

Loosen the grub screw in the knife control knob, and lift the knob clear of the knife shaft.

Loosen all of the M4 screws that hold the top plate to the top of the preparation chamber.

Carefully lift the top plate away from the chamber, applying gentle downward force to the dewar spout and knife shaft, to make sure that you are not putting strain on any of the internal components.

It is likely that the knife shaft will lift out of the prep chamber cold stage but this is normal. Just do not strain anything.
The argon feed capillary tube may be inclined to catch at the back of the chamber so take great care with this.
Place the top plate so that it will not be damaged. You may decide that it is better off upside down.
You will now have access to the internal parts of the chamber.
Using a long M3 Allen key and screw holding tool remove the two screws that retain the SEM side support bracket.

Using a long M2 Allen key, remove the four countersunk screws that hold the cold stage in place on its isolating pillars.

You will now be able to lift out the entire cold stage and dewar assembly but it will still be connected but the electrical leads. Support the stage and dewar assembly while you locate the electrical wire connections in the red, yellow, and black/pink and, with care, disconnect each of the three connections. They pull apart. It should not be necessary to disconnect the white wires as these are for the sputtering shutter solenoid, which will remain in the preparation chamber.

Place the assembly on the clean work place that you prepared earlier.

You will now be able to thoroughly clean the inside of the preparation chamber but take care of the wires from the electrical feedthrough.

Remove the top cold shield from the cold stage taking care no to damage the LED chamber light assemblies.

These LED assemblies are fitted with a glass cover slip that should be cleaned as before, with cotton buds and alcohol.
Thoroughly clean the cold stage taking care not the strain the flexible braids connecting the cold stage to the dewar or the electrical wires to the heater and thermometer.

Re-assembly of the chamber is the reversal of dismantling.

Begin by re-fitting the top cold shield to the cold stage, leading the light wire to the left side of the cold stage so that it will connect to the feedthrough wire set without straining.
Lower the cold stage/dewar assembly into the preparation chamber, connecting the red, yellow and black/pink wires.

Lower the assembly completely into position, engaging the bottom of the dewar into its three locating pegs, and the cold stage onto its four supporting isolation pillars.
Check that the wires are not trapped and moreover that they are not in hard contact with the dewar, cold stage or chamber wall.
Hard contact of the wires between cold and warm surfaces will leak heat into the cold assembly. This must be avoided.

Replace the four countersunk screws into the support pillars through the front window using tweezers, and the two into the SEM side support bracket using the screw retaining tool. Tighten these six screws sufficiently using the correct size Allen key in the way described in the introduction. Do not over tighten any screws.
Carefully inspect the ‘O’-ring in the top edge of the preparation chamber. If necessary, the ‘O’-ring may be removed for cleaning. Removal will not require any tools as it is a loose fit in the ‘O’-ring groove.

Clean the ‘O’-ring using ‘lint free’ tissue and clean alcohol. If the ‘O’-ring is damaged it must be replaced.
Inspect the seal face of the chamber top plate, and clean the seal surface with alcohol.

Place to chamber top plate over the dewar top and lower it down to meet the ‘O’-ring surface. Push the lid forwards so it is aligned with the chamber body and place screws in the two front corner holes, and one in the back centre hole, leaving the screws loose. Place the remaining screws into their holes and progressively tighten them all in rotation sequence until they are all sufficiently tight.

Replace the dewar.

10. Appendix I – The XML control parameters file

All parameters for the control system are saved in an XML file. There are very few hard coded values in the software.

Each parameter has limit values to prevent erroneous values being entered.

The contents of the XML file are described below:

```
<?xml version="1.0" encoding="utf-8"?>
<PP3000TConfig xmlns:xsi=http://www.w3.org/2001/XMLSchema-instance
                                                    xmlns:xsd="http://www.w3.org/2001/XMLSchema">

<SEMWebCamRegistryString></SEMWebCamRegistryString>

        Registry address of SEM web cam

<PrepWebCamRegistryString></PrepWebCamRegistryString>

        Registry address of Prep web cam

<VacuumUnits>MBAR</VacuumUnits>

        Units used to display vacuum readings

<General>

<StartLeakDetectVacuum>4</StartLeakDetectVacuum>

        Vacuum level required to be reached on rotary pump start up (read on airlock Pirani gauge) with no valves
        open; value in millibars

<StartHighVacuumTimeoutSeconds>10000</StartHighVacuumTimeoutSeconds>

        Time allowed for turbo pump to reach required vacuum level before deciding there may be a leak: value in
        seconds

<StartHighOkVacuum>0.0005</StartHighOkVacuum>

        Required turbo vacuum level (read from multi-range gauge on prep chamber) needed to decide system is
        OK; value in millibars

<AirlockVacuumLevel>0.1</AirlockVacuumLevel>

        Vacuum level required to be reached on rotary pump when pumping the airlock (read on airlock Pirani
        gauge); value in millibars

<PumpVacuumTimeoutSeconds>30</PumpVacuumTimeoutSeconds>

        Time allowed for the vacuum to reach the AirlockVacuumLevel when pumping the airlock; value in seconds

<PumpValveOpenTimeoutSeconds>30</PumpValveOpenTimeoutSeconds>

        Time allowed for the user to open the Airlock Valve. If the user is too slow the pumping of the airlock is
        cancelled; value in seconds

<VentAirlockDelaySeconds>30</VentAirlockDelaySeconds>
```

Time allowed for the user to stop pressing the Vent Airlock button. If the user keeps pressing the button, the venting will be cancelled automatically; value in seconds

<SlushDelaySeconds>3600</SlushDelaySeconds>

Time allowed for the user to press the Vent button once the Slush is active. If the user does not press the button in time, the pumping will be cancelled automatically; value in seconds

<TemperatureRampPerMinute>100</TemperatureRampPerMinute>

Requested heating rate for stages; value in degrees per minutes

<TemperatureCoolPerMinute>50</TemperatureCoolPerMinute>

Requested cooling rate for stages; value in degrees per minutes

<BleedVacuumValue>0.05</BleedVacuumValue>

Default setting for the sputter bleed valve vacuum level; value in millibars

<BleedVacuumDelaySeconds>30</BleedVacuumDelaySeconds>

Time to wait before sputter bleed vacuum setting is checked; value in seconds

<BleedVacuumTimeoutSeconds>60</BleedVacuumTimeoutSeconds>

Timeout if sputter bleed vacuum setting is not reached; value in seconds

<CoatingVoltage>600</CoatingVoltage>

Default voltage for sputter coating; value in volts

<CoatingVoltageCheckSeconds>5</CoatingVoltageCheckSeconds>

Time to wait for sputter voltage to rise to set value; value in seconds

<CoatingCurrentCheckLevel>0.5</CoatingCurrentCheckLevel>

Minimum current value to be read from sputter supply to know plasma achieved; value in milliamps

<CoatingVacuumMinimum>0.01</CoatingVacuumMinimum>

Minimum vacuum level for coating to proceed; value in millibars

<CoatingShortCircuitVoltage>80</CoatingShortCircuitVoltage>

Default minimum HT voltage to decide if there is a short circuit on the sputter head (value in volts)

<EvaporationVoltage>10</EvaporationVoltage>

Default evaporation voltage (in volts)

<EvaporationBurnOutSeconds>15</EvaporationBurnOutSeconds>

Time allowed to burn out carbon string (in seconds)

<DegasCurrent>7</DegasCurrent>

Default carbon string degas current (in amps)

<DegasSeconds>20</DegasSeconds>

Default degas time (in seconds)

<LeakDetectionTimeoutSeconds>30</LeakDetectionTimeoutSeconds>

Time allowed during start-up for rotary pump to reach required airlock, buffer tank, or isolated line vacuum level before deciding there may be a leak; value in seconds

<BackingVacuumAutoOn>1</BackingVacuumAutoOn>

Default buffer tank vacuum level (read on buffer tank Pirani gauge) where the buffer tank pump request will be generated; value in millibars

<BackingVacuumAutoOff>0.07</BackingVacuumAutoOff>

Default buffer tank vacuum level (read on buffer tank Pirani gauge) where the buffer tank pumping will stop; value in millibars

<TransferVacuumLevel>0.0001</TransferVacuumLevel>

Default vacuum level where column gate valve can be opened and a specimen transferred (measured on prep chamber multi-range gauge); value in millibars

<PumpLineVaccumLevel>0.02</PumpLineVaccumLevel>

Vacuum level required to be reached when pumping the prep storage (read on airlock Pirani gauge); value in millibars

<AntiConFlowZeroOffset>320</AntiConFlowZeroOffset>

Default offset for anti-con flow gauge calibration (arbitrary units)

<AntiConFlowLitreFactor>0.035</AntiConFlowLitreFactor>

Default gain factor for anti-con flow gauge calibration (arbitrary units)

<SEMFlowZeroOffset>325</SEMFlowZeroOffset>

Default offset for SEM stage flow gauge calibration (arbitrary units)

<SEMFlowLitreFactor>0.038</SEMFlowLitreFactor>

Default gain factor for SEM stage flow gauge calibration (arbitrary units)

</General>

<ComPorts>
<PSUPort>COM3</PSUPort>

COM port used on PC to address the power supply

<AdamPort>COM1</AdamPort>

COM port used on the PC to address the ADAM modules

</ComPorts>

<PrepVacuum>
<MinVacuum>1E-06</MinVacuum>

Lowest value displayed on the prep vacuum analogue scale; value in millibars

<MaxVacuum>0.01</MaxVacuum>

Highest value displayed on the prep vacuum analogue scale; value in millibars

</PrepVacuum>

<AirlockVacuum>
<MinVacuum>0.001</MinVacuum>

Lowest value displayed on the airlock vacuum analogue scale; value in millibars

<MaxVacuum>1000</MaxVacuum>

Highest value displayed on the airlock vacuum analogue scale; value in millibars

</AirlockVacuum>

<Anticontaminator>

<Temperature>
<MinTemperature>-196</MinTemperature>

Lowest temperature which can be entered by the anti-contaminator controls; value in degrees Centigrade

<MaxTemperature>30</MaxTemperature>

Highest temperature which can be entered by the anti-contaminator controls; value in degrees Centigrade

<InitialTemperature>-175</InitialTemperature>

Default temperature setting for the anti-contaminator; value in degrees Centigrade

<HeaterGain>0.5</HeaterGain>

Default heater gain for anti-con (arbitrary units)

<HeaterDerivative>0.5</HeaterDerivative>

Default heater derivative for anti-con (arbitrary units)

<HeaterIntegral>0</HeaterIntegral>

Default heater integral for anti-con (arbitrary units)

</Temperature>

<GasFlow>
<MinGasFlow>0</MinGasFlow>

Lowest gas flow which can be entered for the anti-contaminator; value in litres/minute

<MaxGasFlow>5</MaxGasFlow>

Highest gas flow which can be entered for the anti-contaminator; value in litres/minute

<InitialGasFlow>0</InitialGasFlow>

Default gas flow for the anti-contaminator; value in litres/minute

<GasFlowGain>0.02</GasFlowGain>

Default gas flow gain (arbitrary units)

<GasFlowDerivative>0.3</GasFlowDerivative>

Default gas flow derivative (arbitrary units)

<GasFlowIntegral>0.05</GasFlowIntegral>

Default gas flow integral (arbitrary units)

</GasFlow>

</Anticontaminator>

<SEMCryoStage>

<Temperature>
<MinTemperature>-196</MinTemperature>

Lowest temperature which can be entered by the SEM stage controls; value in degrees Centigrade

<MaxTemperature>50</MaxTemperature>

Highest temperature which can be entered by the SEM stage controls; value in degrees Centigrade

<InitialTemperature>-140</InitialTemperature>

Default temperature for the SEM stage; value in degrees Centigrade

<HeaterGain>0.1</HeaterGain>

Default heater gain for SEM stage (arbitrary units)

<HeaterDerivative>0.1</HeaterDerivative>

Default heater derivative for SEM stage (arbitrary units)

<HeaterIntegral>0.1</HeaterIntegral>

Default heater integral for SEM stage (arbitrary units)

</Temperature>

<GasFlow>
<MinGasFlow>0</MinGasFlow>

Lowest gas flow which can be entered for the SEM stage; value in litres/minute

<MaxGasFlow>5</MaxGasFlow>

Highest gas flow which can be entered for the SEM stage; value in litres/minute

<InitialGasFlow>0</InitialGasFlow>

Default gas flow for the SEM stage; value in litres/minute

<GasFlowGain>0.02</GasFlowGain>

Default gas flow gain (arbitrary units)

<GasFlowDerivative>0.15</GasFlowDerivative>

Default gas flow derivative (arbitrary units)

<GasFlowIntegral>0.05</GasFlowIntegral>

Default gas flow integral (arbitrary units)

```
</GasFlow>

</SEMCryoStage>

<PrepChamber>
<MinTemperature>-196</MinTemperature>
```

Lowest temperature which can be entered by the Prep stage controls; value in degrees Centigrade

```
<MaxTemperature>50</MaxTemperature>
```

Highest temperature which can be entered by the Prep stage controls; value in degrees Centigrade

```
<InitialTemperature>-140</InitialTemperature>
```

Default temperature for the prep stage; value in degrees Centigrade

```
<HeaterGain>0.5</HeaterGain>
```

Default heater gain for prep stage (arbitrary units)

```
<HeaterDerivative>0.3</HeaterDerivative>
```

Default heater derivative for prep stage (arbitrary units)

```
<HeaterIntegral>0.1</HeaterIntegral>
```

Default heater integral for prep stage (arbitrary units)

```
</PrepChamber>

<SavedCoatingRecipes>
<string></string>
<string />
<string />
```

Names of the saved sputter coating recipe files

```
</SavedCoatingRecipes>

<SavedSublimeRecipes>
<string></string>
<string></string>
<string />
```

Names of the saved sublimation recipe files

```
</SavedSublimeRecipes>

<SavedEvaporationRecipes>
<string xsi:nil="true" />
<string xsi:nil="true" />
<string xsi:nil="true" />
```

Names of the saved evaporation recipe files

```
</SavedEvaporationRecipes>

</PP3000TConfig>
```

11. Appendix II – Service and diagnostics mode

11.1. Accessing the configuration and diagnostic modes

Touching the logo at the lower left of the screen opens a fly out menu.

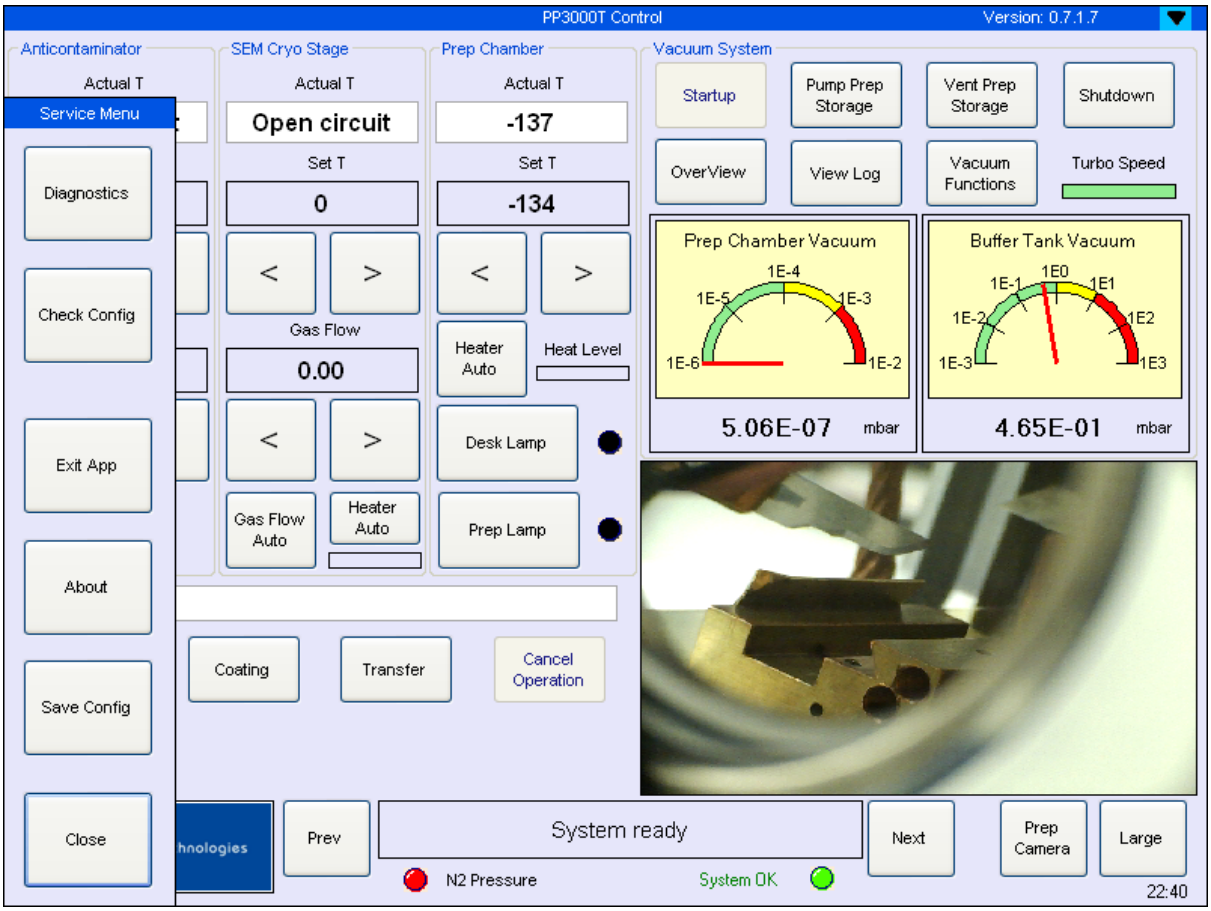
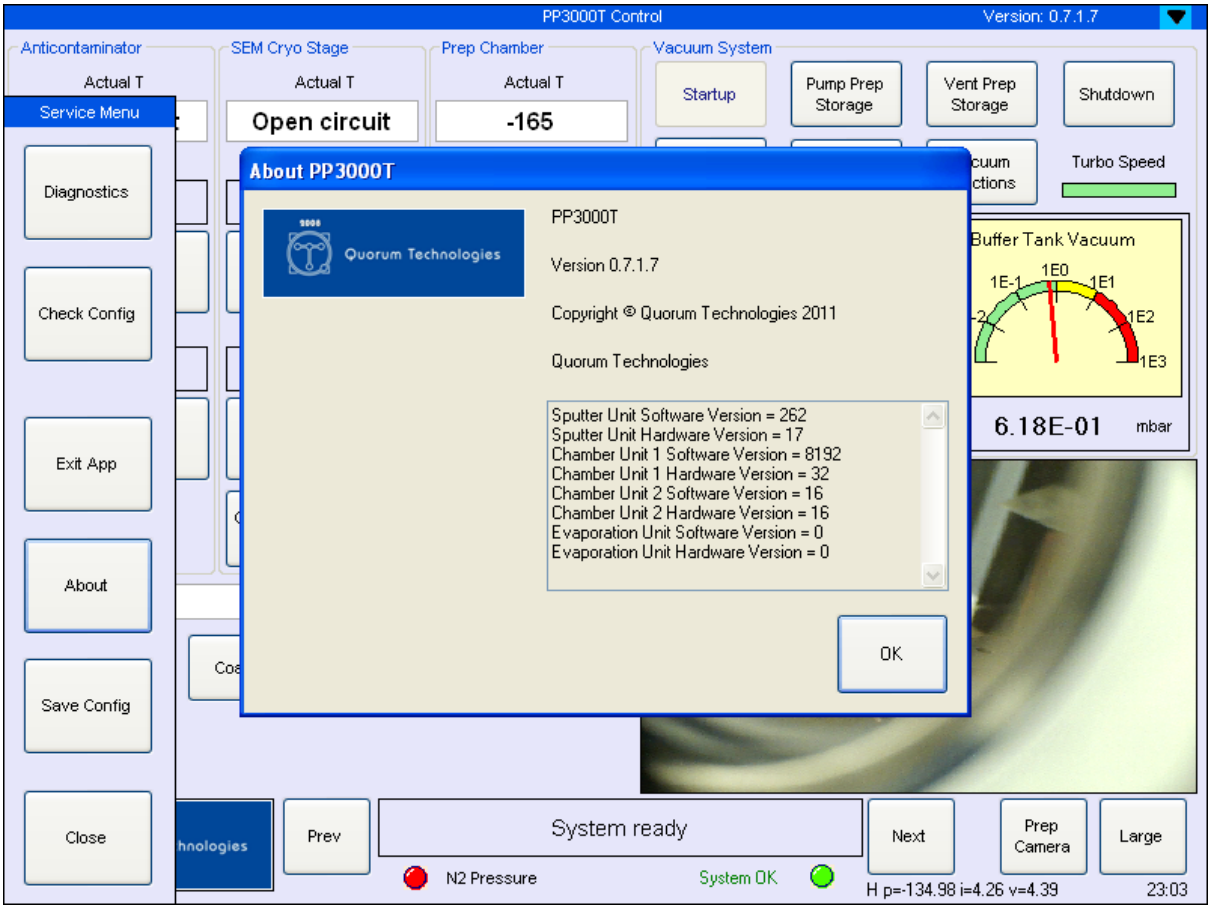


Figure 87 - Fly out menu

The “Exit App”, “About” and “Save Config” buttons are not password protected.

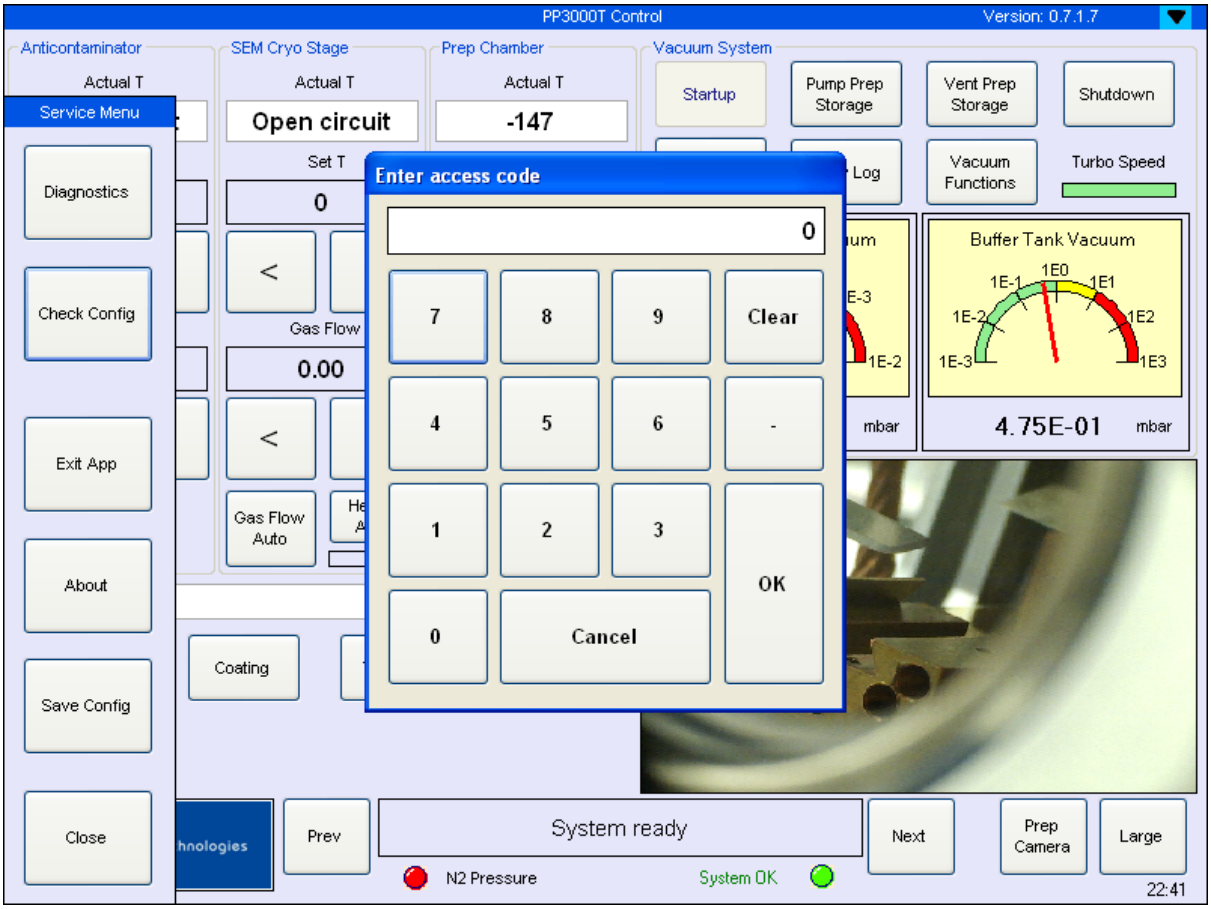
“Exit App” closes down the software.

“About” brings up a small window with details of the system.



"Save Config" writes any new parameters which have been set in the configuration to the XML file.

To access the first 2 menu items the user must supply an access code.



Typing in the correct code will open either of the 2 windows. These 2 modes are not intended for normal usage as it is possible to cause damage to the system by entering incorrect values.

11.2. Configuration mode

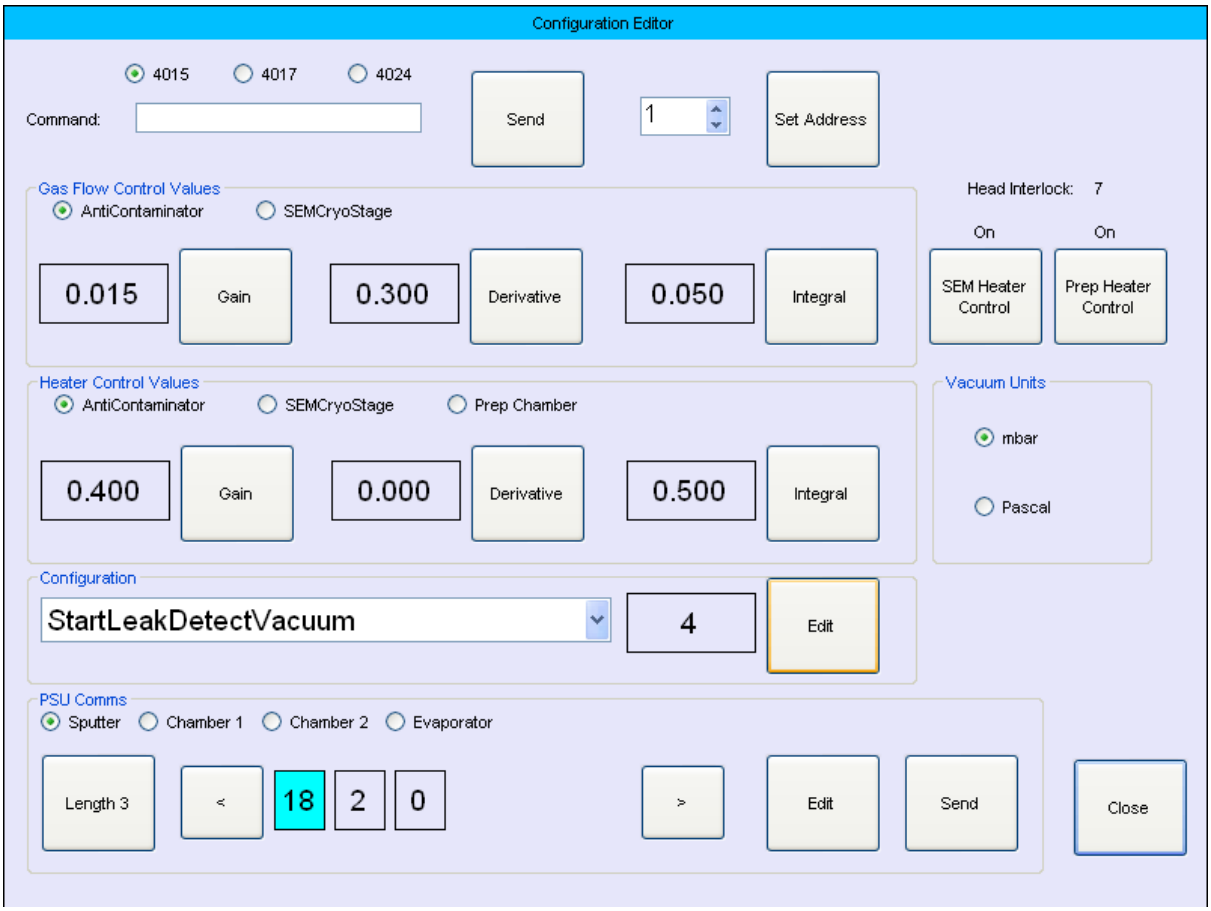


Figure 88 - Configuration page

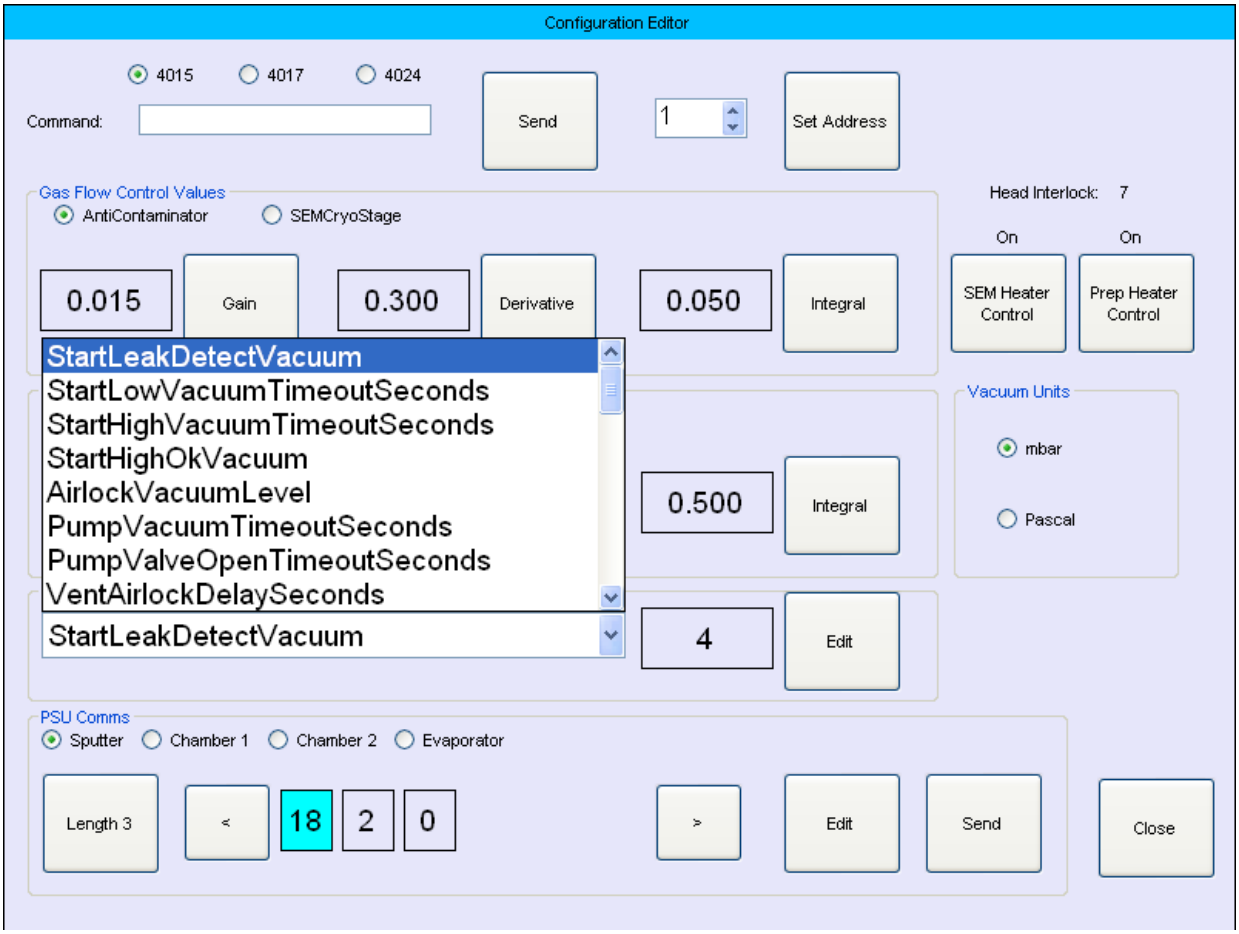


Figure 89 - Configuration drop down menu

11.3. Diagnostics mode

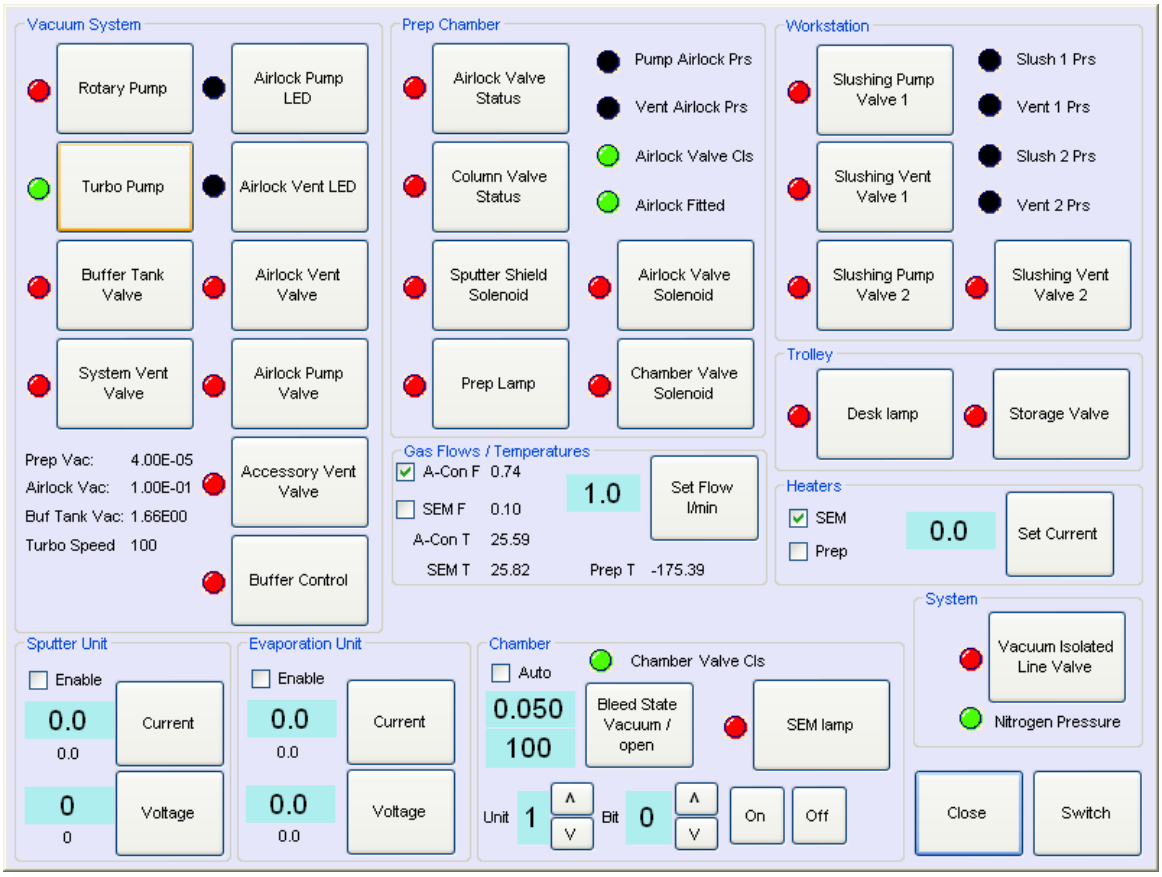


Figure 90 - Diagnostics page

12. Glossary of terms

Buffer tank

In a standard vacuum pump configuration, a rotary pump is used to provide a backing vacuum to a turbo molecular pump. The rotary pump runs continuously. In electron microscopy applications the vibrations from the rotary pump could be transmitted to the microscope. To avoid this, a vacuum reservoir is used between the rotary pump and the turbo pump. This reservoir is called a buffer tank and is pumped to a low vacuum by the rotary pump and then a valve between the rotary pump and buffer tank is closed and the rotary pump turned off. A Pirani gauge is then used to monitor the vacuum level in the buffer tank and when this rises to a pre-set value the rotary pump is started again and the valve opened to re- evacuate the buffer tank. The rotary pump then only runs for a few minutes each hour.

Sublimation

Cryo electron microscopy (Cryo-SEM)

Cryo-SEM is a method for preparing liquid, semi-liquid or beam-sensitive specimens for examination in a scanning electron microscope (SEM). The technique has huge benefits compared to alternative methods, such as critical point drying and freeze drying, because all of the specimen's water is retained.

Cryo transfer

Cryo transfer device

Slusher Pot

Sputter coating

Magnetron sputtering using a crossed-field electromagnetic configuration keeps the ejected secondary electrons near the cathode (target) surface and in a closed path on the surface. This allows a dense plasma to be established near the sputter target surface. The ions that are accelerated from the plasma do not sustain energy loss by collision before they bombard the sputter target.

For electron microscopy (EM) specimen coating, the magnetron sputtering head design ensures that minimal heat energy (electrons) reach the specimen surface. This is important as it reduces heat damage to specimen and is a significant factor in ensuring the grain size within the sputtered film is optimally small – essential for high resolution field emission scanning electron microscopy (FE-SEM).

Sputter coating with metals such as platinum, gold-palladium or chromium is widely used for depositing fine grain, high-resolution films onto field emission scanning electron microscopy (FE-SEM) specimens. Chromium oxidises on contact with air, which can present specimen storage problems. For this reason, iridium (Ir) sputter coating is increasingly preferred by many workers.

Fracturing

Carbon coating

The thermal evaporation of carbon (C) is widely used for preparing specimens for electron microscopy (EM). A carbon source - either in the form of woven fibre or graphite rod - is mounted in a vacuum system between two high-current electrical terminals. When the carbon source is heated to its evaporation temperature, a fine stream of carbon is deposited onto specimens.

The main applications of carbon coating in EM are making scanning electron microscopy (SEM) specimens conductive for subsequent examination by X-ray microanalysis, and being used as specimen support films on transmission electron microscopy (TEM) grids.

Carbon fibre

Carbon fibre, normally in the form of a woven cord, can be used to thermally evaporate thin layers of carbon onto a substrate. The main application in electron microscopy (EM) is the production of thin, electrically-conducting coatings on scanning electron microscopy (SEM) specimens. Carbon fibre can be used for transmission electron microscopy (TEM) applications, but carbon rod is normally preferred due to superior control of the evaporation process.

Film thickness monitor

A film thickness monitor can be used to monitor and control the thickness of sputtered and evaporated metal films. A gold-coated quartz crystal is mounted in the vacuum chamber of the coating system, ideally close to the specimen or substrate. The quartz crystal is made to oscillate at a defined frequency, using an externally-mounted oscillator. As metal is deposited on the quartz crystal, the frequency of oscillation alters and the change is converted to a digital (eg LED) display on the monitoring unit.

Shuttle

In this context a shuttle is the dovetail shaped, gold-plated copper block which is used to transfer specimens or specimen stubs between different parts of the system. It fits onto the end of the insertion rod via a bayonet fitting.

Stub

A stub is a cylindrical metal disc usually 10mm diameter and between 5mm and 10mm thick. It may be made of copper or aluminium for good thermal conductivity. Specimens are mounted onto these stubs which can have multiple holes and slots machined in them.

Preparation chamber

The preparation chamber is a vacuum chamber mounted on the side of the SEM. It has an integral cold stage and anti-contaminator as well as coating and fracturing facilities. This is where all cryo specimens will be processed before transfer into the SEM.

Penning gauge

Penning gauges are ionization gauges which operate with a cold discharge (cold-cathode). The discharge which takes place in a measuring tube is very similar to that of a sputter ion pump. The gauge has two electrodes (cathode and anode) between which a discharge is struck and maintained by a high DC voltage (approx 2 kV). A magnetic field is applied to make the paths along which electrons sufficiently long that their collision rate with gas molecules is large, creating a large number of ions required to maintain the discharge. The magnetic field lines confine the electrons to a spiral path. The ions produced by these collisions move to the electrodes and form a pressure-dependent discharge current. The value current is dependent on the type of gas molecules present in the vacuum.

Pirani gauge

Pirani gauges are gauges for measuring vacuum that use a heated sensor wire. Pressure is determined by measuring the current needed to keep the wire at a constant temperature. A Pirani gauge will only measure vacuum levels in the lower range (down to 10^{-3}mbar) and is typically used in low vacuum systems, such as rotary-pumped sputter coaters.

A Pirani gauge is sometimes used in conjunction with a high vacuum gauge, such as Penning, to measure vacuum in the low pressure range. Most of our high vacuum products now use full-range gauges - a single gauge which measures vacuum from atmosphere to high vacuum.

Rotary pump

A rotary pump is a type of vacuum pump where pumping is produced by moving air from one side of a rotating cylinder to another by means of an eccentric drum. Rotary pumps are used with all of our coaters, either to evacuate instruments directly, or to ‘rough pump’ and ‘back’ high-vacuum sputter coaters and vacuum evaporators.

Turbo pump

A turbo-molecular (turbo) pump is a type of vacuum pump used to obtain and maintain high vacuum. The principle of operation is that gas molecules within a vacuum chamber can be given momentum in a desired direction by repeated collisions with a rapidly spinning turbine rotor. The rotor ‘hits’ gas molecules from the inlet of the pump towards the exhaust in order to create or maintain a vacuum. A turbo pump normally works in tandem with a low-vacuum pump, such as a rotary vacuum pump, which is used to ‘rough pump’ the vacuum system (e.g. sputter coater or vacuum evaporator) during initial pump-down period, and to ‘back’ the turbo pump (i.e. remove gases from the back of the pump) during high-vacuum operation.

Liquid nitrogen freezing

Using liquid nitrogen is the cheapest and easiest way to prepare cryo SEM specimens. Normally, the specimen is plunged rapidly under slushed nitrogen (see Liquid nitrogen slush). Although the freezing is not that rapid, it is usually enough for cryo-SEM.

Liquid nitrogen slush

When pumped by a rotary vacuum pump, liquid nitrogen can be solidified. Liquid nitrogen freezes at 63 K (−210 C). The freezing efficiency of liquid nitrogen is severely limited by the fact that it boils immediately on contact with a warm object. This creates an insulating nitrogen gas layer around the object. This effect is known as the Leidenfrost effect. By plunging an object into slushed nitrogen more rapid cooling may be obtained than plunging into liquid nitrogen at its boiling point (77K, -196 C).

Rivets

Rivets are used for liquid specimens and rapid freezing. Two rivets are sandwiched together and plunged into nitrogen slush. Their thin wall section and small volume give good freezing rates. The rivets are then mounted in a stub and transferred to the preparation chamber where the upper rivet if knocked off to reveal the fractured surface.

Anti-contaminator

SEM cold stage

Flushing

Heat exchanger

Argon gas

Argon (Ar) is widely used as the process gas during sputter coating. The use of argon is essential for clean, contamination-free coating of scanning electron microscopy (SEM) specimens.

Mounting media

In order to attach a specimen to a shuttle or stub some sort of “glue” is needed. The glue should not shrink when frozen nor bubble nor crack. The usual media used for cryo-SEM is a 1:1 mixture of Tissue-Tek® and colloidal graphite.

13. Circuit Diagrams